

LABORATORY STUDIES IN CHEMISTRY

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BRADBURY

GENERAL SUGGESTIONS TO THE STUDENT

1. Solids — like match sticks, bits of glass or metal — should never be thrown into the sinks. They clog the plumbing and cause great annoyance and damage. Put them in the waste jars provided for the purpose.

2. Paper is out of place in the sinks. It is dangerous to put it in the waste jars, where it often takes fire. The proper place for it is the waste-paper basket.

3. The student will find that he has a strong tendency to take *too much* of the various substances required in his work. This tendency must be overcome before any real progress can be made. The quantity needed is stated in each study. To take more means waste of material and of time.

4. Never pour water into concentrated sulphuric acid. In diluting sulphuric acid, the acid should be added to the water in a thin stream, with constant stirring.

5. When it is necessary to mix ammonia with an acid, both should be diluted before mixing. Explosive interaction occurs when concentrated ammonia is mixed with concentrated acids.

6. Before using a vessel, make sure that it is *clean*. Experiments in dirty vessels are not worth making, for the results are only misleading.

7. Learn to regulate the *height* of your burner flame. For heating test tubes and beakers, a small flame is better than a large one.

8. Take at least five minutes at the end of each laboratory period to clean up your glassware and put your working place in order.

THE NOTEBOOK

9. Neat, full, and accurate notes are necessary, especially to those of you who expect to obtain credit for your chemical work in college, university, or before any examining board. *Do not copy the directions from the manual.* State, in your own way, the object of each experiment, the method and apparatus, the results, and your conclusions. Frequently, a simple line drawing, similar to those in the textbook, will make your description easier to understand. The notebook should be provided with an index.

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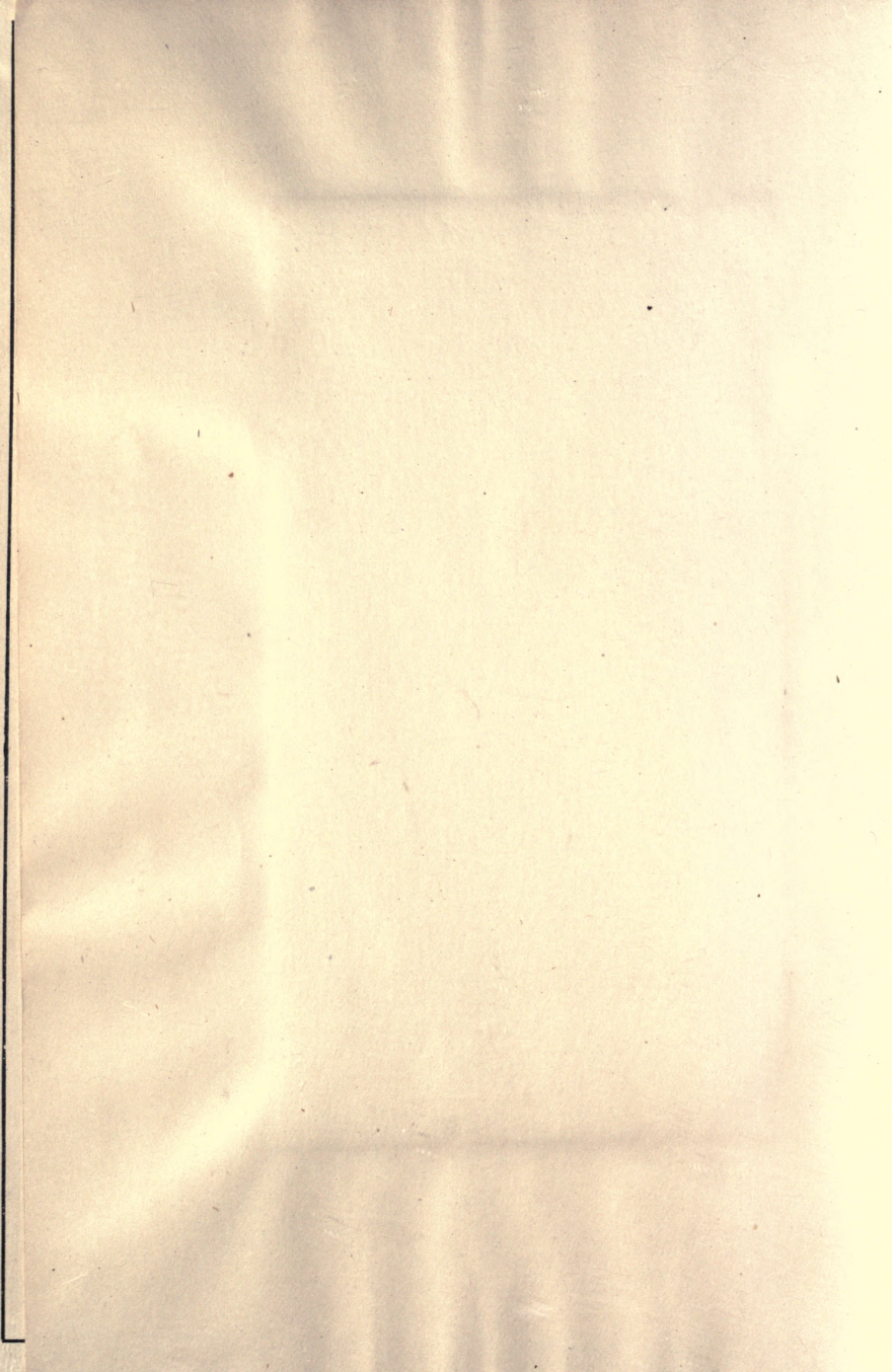
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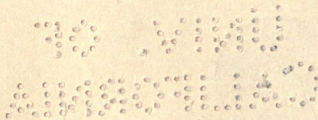
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PREFACE

Briefly stated, the main features of this laboratory manual are as follows:—

1. It covers thoroughly the various syllabi which teachers preparing students for college have to consider.

2. Each exercise, including the writing of the notes, can be finished within ninety minutes.

3. The apparatus required is simple and inexpensive, and due regard has been given to the prices of the chemicals. Expensive substances are employed at times, but only in very small quantities.

4. The fact has not been overlooked that the teacher of science usually carries as many hours on the roster as the teacher who has no apparatus or materials to arrange. I have tried to simplify the manual labor required of the teacher, without reducing the efficiency of the work.

5. The experienced teacher will at once perceive that the practical details of the experiments have been worked out with unusual care. It has, in fact, taken me many years to get these laboratory studies into the shape in which they are here presented, and I have refrained from publishing them until I had made sure that they were as perfect as the painstaking labor of my students and myself could make them. I shall be very grateful indeed for any suggestions as to further improvements from other teachers.

It is, of course, as impossible as it is undesirable to frame a set of directions which shall be a substitute for the teacher. What I have tried to do is merely to save the appalling waste of energy involved in the constant repetition of details such

as quantity needed, vessel to use, height of flame, dilution, etc. To supply this sort of information is precisely the function of the laboratory manual.

6. It is not at all difficult, at present, to put together a set of exercises which will occupy the laboratory time of the beginner for a year in a pleasant and orderly way. It is less easy to make sure that each experiment is significant and worth while, that it adds something to the logical development of the student's ideas and to his appreciation of the general points of view of the science. I confess that much that is offered under the caption of laboratory work seems to me hardly worth the time it takes. The student who comes to the laboratory might otherwise be doing serious work of permanent value in languages or in mathematics. His time should not be wasted with purely spectacular and pyrotechnic experiments, or with so-called quantitative studies, —like the heating of magnesium powder— in which even the most finished experimental technique must fail to secure reasonably exact results.

One of the chief devices I have employed in attempting to give greater educational value to the work is *classification*. For instance, the facts which relate to some central topic such as the action of soluble hydroxides on salt solutions, or the effect of heat upon carbonates, are not scattered through the book in a wholly aimless way: they are organized into a compact laboratory exercise which serves, once for all, to make the matter clear. Examples of this general principle of arrangement will be found in the exercises numbered 14, 22, 31, 37, 58, 59, and others.

7. Probably all teachers agree, nowadays, that some of the work should be *quantitative*. The quantitative experiments in this book have been worked out with the utmost care. Since I have, in my notebooks, upwards of a thousand results for each of them, I may fairly say that they are quite within the capacity of high school students, who, in fact,

work them out with enthusiastic interest. Naturally, the effect in improving the manipulative technique of the students is very marked. Far more important is the reality which is given to their conceptions of the foundations of the science. It is almost impossible to teach quantitative ideas without quantitative experimentation to give substance to the teaching.

A balance sensitive to a milligram can now be bought for about ten dollars. Small weights, which may be lost from the set, can be easily and cheaply replaced by cut lengths of No. 36 German silver wire, B. & S. gauge, one centimeter of which weighs almost exactly one milligram. The rest of the apparatus required is of the simplest description.

8. The literature of the elementary laboratory is now quite extensive, and I have tried to make a careful survey of it. Among the books which have been most helpful are Stoddard's *Quantitative Experiments in General Chemistry*, Karl Scheid's *Praktischer Unterricht in Chemie*, Ohmann's *Chemie*, and the Laboratory Manual of Alexander Smith.

The quantity of material required in the experiments is, in all cases, stated in a precise numerical way. These statements are intended simply to give the student a good *approximate idea* of the amount he should take. It is only in the *quantitative* exercises that the quantity stated should be accurately weighed or measured. The apparatus and materials needed are stated at the beginning of each exercise. Supplies always on the student's table, such as the burner, the stand, and the ordinary acids, are omitted.

A lead disk about 6×2 cm., pierced radially and axially is convenient for collecting gases over water. It is used in an agate pan, the bottle of water being inverted over the central perforation. The disk also has been omitted from the lists of necessary supplies, since there are many other devices which may serve the purpose equally well.

It may not be amiss to call the attention of teachers to the use of potassium permanganate, instead of manganese dioxide,

for generating chlorine, and of formic acid, instead of oxalic acid, in the preparation of carbon monoxide. In both cases, the older methods have been obsolete among chemists for some years, and one cannot help wondering at the misplaced conservatism which causes their continued republication in the newer elementary texts.

It is not improbable that chemists who are not engaged in secondary teaching may be surprised at the care which has been taken to exclude complex and expensive apparatus. Let me add, therefore, that the secondary school teacher handles sections of thirty or more without laboratory assistance and without time for preparation, that he must finish his exercise in a definite time, which is usually not greater than ninety minutes, and that his students have had no opportunity to acquire manipulative skill. When these conditions are generally understood, a certain amount of energy, which is now wasted in rather futile criticism of secondary aims and methods, will be expended in some more useful way. In fact, to do the best that can be done under these circumstances forms a difficult problem, which is quite different from the problem of the college or university instructor. To offer, in a small way, some help toward the solution of the problem of elementary teaching is the object of the present book.

Professor Alfred L. Carey of the Southern High School has assisted me with the reading of the proof. The illustrations are from original drawings made by my wife.

ROBERT H. BRADBURY.

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LABORATORY STUDIES IN CHEMISTRY

EXERCISE 1

GLASS ROD. TUBING

Apparatus. — Triangular file. Bunsen burner. Wing-top.

Materials. — Glass rod and tubing. Sugar.

Method. — Glass rod or tubing can be cut by making a notch with a file and bending away from the notch.

A. Glass rod. — With a triangular file, make a notch on a piece of thin glass rod 15 centimeters (6 in.) from the end.¹ One sharp stroke of the file is sufficient. Holding the rod as indicated in Fig. 1, endeavor to bend it away from the notch, and it will break off at that point. Cut off three such pieces. Since the ends of the rods are jagged and inconvenient to handle, round both ends of each rod by holding it in the Bunsen flame and rotating the rod gently. Support the rods by the middle on your test-tube rack until they cool. (Hot glassware or hot apparatus of any kind must never be laid on the desk or put away under it.)

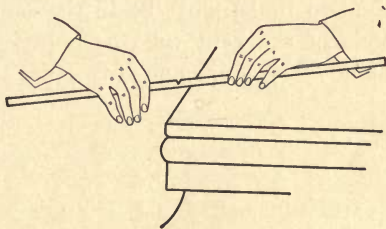


FIG. 1.

B. Glass tubing. — Cut, just as you cut the glass rod, a piece of glass tubing 20 cm. (8 in.) in length. Hold the middle of the piece of glass tubing in the flame, turning it slowly, and

¹ Rod not over 3 millimeters in diameter is best. The use of thick stirring-rods is a frequent cause of breakage of beakers.

when it becomes red-hot, gently and slowly draw the two portions apart. Do not twist the tubes. The pull must be straight. Let the two tubes cool and use them to study the effect of heat upon a fragment of wood (match-stick) and a little sugar.

Bending glass tubing.—For bending, fit a wing-top on the Bunsen burner. Hold the tube so that the flame heats as long a portion as possible, and rotate it so that it is evenly heated. When sufficiently hot, remove it from the flame and make the bend.

In this way, bend a glass tube 15 cm. (6 in.) long into an acute angle (Fig. 2). Cut a piece of glass tubing about 25 cm.



FIG. 2.



FIG. 3.

(10 in.) long and bend it twice at right angles (Fig. 3).

This must be done so that, when the double bend is laid upon the table, every part of it will touch the surface of the latter, or, in other words,

the two limbs must be in the same plane. Round the sharp ends of the bent tube by holding them a short time in the flame—not long enough to cause them to collapse.

EXERCISE 2

CAPACITY OF LABORATORY VESSELS

Apparatus. — A 100 cubic centimeter cylinder, graduated in cubic centimeters. (Use the expression c.c. for cubic centimeter.) Large and small test tubes. Evaporating dish. Bottle. Beaker.

Materials. — Rubber bands.

Method. — The vessel is filled with water, which is then poured into a graduated cylinder.

Measure 5 c.c. of water in the graduated cylinder and pour it into the *small* test tube. This tube is 6 in. long by $\frac{3}{4}$ in. diameter. In metric measure it is 15 cm. by 1.8 cm. Mark the upper level of the water by a rubber band slipped around the tube. See that the upper edge of the band is at the lower meniscus of the water (Fig. 4). Memorize the appearance of 5 c.c. of water so that when you are asked to take that quantity of a liquid in an experiment you may have a definite idea of how much is meant. Add another 5 c.c. of water, mark the upper level, and memorize the appearance of 10 c.c. of liquid. Now fill the tube completely and pour the water into the empty measuring cylinder.

The length of the *large* test tube is 8 in. (about 20 cm.) and its diameter 1 in. (2.5 cm.). Repeat the work, using the large test tube. Where the capacity of a vessel is greater than 100 c.c., determine it by first pouring exactly 100 c.c. of water in it and then measuring the additional water required to fill it.

Place first 25 c.c. and then 50 c.c. of water in the beaker, marking the levels. Then get the total capacity. Get the total capacity of your evaporating dish and one of your bottles. Record the results.



FIG. 4.

EXERCISE 3

DENSITY OF LIQUIDS

Apparatus. — Trip scales and weights. 100 c.c. measuring cylinder graduated in c.c.

Materials. — Water. 95 per cent grain alcohol. Saturated salt water.

Method. — A measured volume of the liquid is weighed.

Clean the cylinder if necessary and dry it carefully with a towel. Weigh it as accurately as possible and record the weight in your notebook. Count the weights at least twice.

Remove the cylinder from the scales and place in it 20 c.c. of water. Read from the bottom of the meniscus (Fig. 5).

Weigh the cylinder with the water. Get the weight of the water by difference, and calculate the weight of 1 c.c.

Is the result exactly what you would expect? If not, what is the probable reason? Bearing in mind the limits of accu-

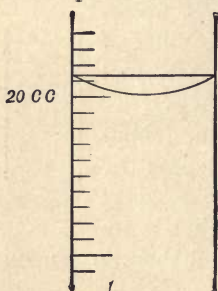


FIG. 5.

racy of the scale and the cylinder, how exact would the result be, provided that the work was done as well as possible? Would the fourth decimal place have any value? the third? the second? Why?

Dry the cylinder inside and out. You need not weigh it again. Place in it 20 c.c. of alcohol and get the weight of 1 c.c. in the same way. Look up the density of alcohol and see how close your result is to the truth.

In the same way, determine the weight of 1 c.c. of saturated brine.¹

¹ The weight of 1 c.c. of saturated brine is about 1.2 grams.

EXERCISE 4

DENSITY OF SULPHUR, COPPER SULPHATE, AND MARBLE

Apparatus.—Trip scales. Graduated cylinder. Evaporating dish.

Materials.—Broken roll sulphur, free from dust. Clean marble chips. Copper sulphate crystals (commonly called *bluestone*). Salt. Alcohol.

Method.—The volume of a weighed portion of the solid is obtained by dropping it into water in a graduated cylinder.

Weigh or counterpoise the dish. Weigh in it exactly 10 grams of fragments of sulphur, free from dust. The cylinder need not be weighed. Place in it exactly 20 c.c. of water. Incline the cylinder and carefully slide in the 10 grams of sulphur. The increase in volume is the volume of the sulphur. Calculate the weight of 1 c.c. of sulphur. Look up the density of sulphur and see how nearly right you are.

Dry the wet sulphur as well as you can on paper, and put it in the place designated by the instructor. Repeat the whole experiment, using marble chips instead of sulphur. Dry the marble as well as possible and place it in the receptacle provided.

Since bluestone dissolves in water, you must use some other liquid. Weigh off 10 grams of bluestone, just as you did with the others, but place 20 c.c. of *alcohol* in the dry cylinder. Otherwise, the determination is made in the same way as the other two. Return the alcohol to the bottle and place the bluestone in the receptacle provided.

If time permits, determine the density of solid salt just as you did that of bluestone, using alcohol because salt is soluble in water. The salt may be thrown into the waste jar. The alcohol should be returned to the bottle.

EXERCISE 5

SULPHUR

Apparatus. — Watch glass. Agate pan. Lens. Funnel. Two small dry test tubes.

Materials. — Roll sulphur. Powdered roll sulphur. Flowers of sulphur. Carbon disulphide. Filters.

Method. — See textbook, pages 4, 5, 6, and 7.

CAUTION: *Carbon disulphide is highly inflammable. It must not be heated nor used near a flame.*

Examine roll sulphur. Is it brittle or malleable? Dense or light? Rub a piece on the coat sleeve, and bring it near very small pieces of paper. What is the result? Does it dissolve in water? (Use a small piece.)

Examine flowers of sulphur and record its properties.

(a) Place 0.5 c.c. of *powdered roll sulphur* in a dry, small test tube and add 2 c.c. of carbon disulphide. Shake until the sulphur dissolves. Put the liquid into a watch glass and let it evaporate in a place *as far from the flame as possible*. Examine the crystals with a lens. Make drawings of several crystals. These crystals are sulphur. This form of sulphur is called α -sulphur.¹

(b) Have ready a dry filter folded as though to go into a funnel (Fig. 6) and a pan of water. Half fill a small dry test tube

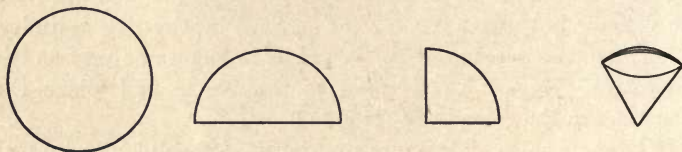


FIG. 6.

with crushed roll sulphur. Hold the tube with a holder or clamp and melt the sulphur by holding the tube above a small flame and turning it constantly. The melted sulphur should be pale amber-yellow.

¹ α (Alpha) = first letter of Greek alphabet.

Hold the filter by the edge of the threefold portion and pour the melted sulphur into it. Observe the crystallization of the sulphur. When crystals begin to form across the surface, pour out the liquid portion into the pan of water. Unfold the filter at once. Make drawings of several crystals as they appear when viewed with a lens. Compare with the form of the crystals obtained in (a). Is the color of the two forms of sulphur the same? Are both forms equally brittle?

The form of sulphur you have just made is called β -sulphur.¹ Set some of the crystals aside for twenty-four hours, and explain the change which occurs.

Examine the sulphur which has been poured into the water. Break a piece of it. Is it brittle or soft? Does it probably consist of α -sulphur or β -sulphur? Of which would it consist if you kept it for a day or two?

(c) Half fill the same test tube again with crushed roll sulphur and melt cautiously as before. Hold the tube with a holder or take the clamp from your stand. This time continue heating, studying the changes in the color and *viscosity* (thickness) of the liquid. When the sulphur boils, light the vapor at the mouth of the tube and pour the liquid slowly into the pan of water, moving the tube in a circle around the funnel so as to obtain a coiled thread of sulphur, rather than a lump (Fig. 7).

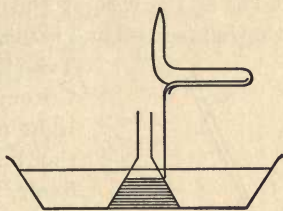


FIG. 7.

Examine the product. Is it hard or soft? Is it elastic? Does it resemble the product formed when melted sulphur was poured into water in (b)? Keep some of the product from (c) and describe any change which takes place in it. Is it a stable form of the element? Should you expect to find it in nature?

¹ β (Beta) = second letter of Greek alphabet.

EXERCISE 6

GALENITE. THE USE OF THE BLOWPIPE

Apparatus.—Blowpipe. Wing-top burner. Funnel. Small test tube.

Materials.—Asbestos fiber. Prismatic blowpipe-charcoal. Galenite (a crystallized specimen for examination and some of the crushed mineral). Minerals can be crushed with a hammer upon a heavy iron plate. A cylinder of tin or cardboard, about 8 cm. high \times 15 cm. in diameter, open at both ends, is convenient to prevent the mineral from flying sideways.

Method.—See textbook, page 10.

(a) Wash off the mouthpiece of the blowpipe. Close the holes at the base of the burner and fit on the wing-top. Use the *luminous* flame turned down to a height of 2.5 cm. (1 in.).

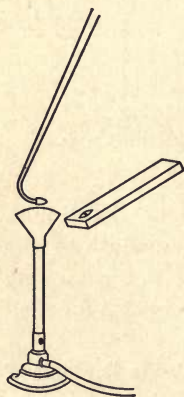


FIG. 8.

Take the blowpipe with the right hand about 7.5 cm. (3 in.) above the bend. Rest the right elbow on the table and put the tip of the blowpipe about halfway through the flame, the broad surface of which should be toward you. Blow gently. Keep the cheeks somewhat distended. Use them as a reservoir of air which is replenished when necessary from the lungs. The natural tendency of the cheeks to collapse will give sufficient blast. The flame should be blue, noiseless, and steady. A flickering or noisy flame indicates that the blast is too strong.

(b) Make, with your knife, a depression about 3 mm. (0.1 in.) deep by 1 cm. (0.4 in.) diameter, near one end of the charcoal. Place a 2 mm. (0.08 in.) fragment of galenite in this depression and direct the flame upon it, holding the charcoal sloping upward away from the flame at an angle of about 20° . The flame should be in the same plane as the long axis of the coal (Fig. 8). The hottest part of the flame is just beyond the tip of the inner blue cone.

When the action seems complete, let cool, remove the product from the charcoal, and examine. Is it brittle, like galenite? What is it?

CAUTION: Avoid inhaling the red poisonous gas which escapes when nitric acid acts upon metals or other substances.

(c)¹ Place 1 c.c. of powdered galenite in a small test tube and add 2 c.c. of concentrated nitric acid. Warm gently with a small flame, not touching the tube, until the mineral disappears. Half fill the tube with water. Plug up the angle of a funnel with asbestos and pour the liquid upon it. With the aid of more water, get all the material in the tube upon the asbestos. Wash three times with water to remove nitric acid. What does the residue appear to be? Let it dry as long as time will permit, place it on charcoal, and heat with the blowpipe flame. When it begins to burn, stop heating and notice the odor. Does this confirm your guess as to its nature?

EXERCISE 7

PYRITE

Apparatus.—Blowpipe. Wing-top burner. Small test tube. Funnel. Magnet.

Materials.—Pyrite (a well-crystallized specimen for examination and some of the crushed mineral for class use). Asbestos. Prismatic blowpipe-charcoal. Glass tubing.

Method.—See textbook, page 13.

(a) Place a 2 mm. (0.08 in.) fragment of pyrite in a depression on charcoal and heat it in the same way as the galenite. Interrupt the heating from time to time to notice the odor produced. What element is indicated by this? When the action seems complete, break up the residue and try the action of a magnet upon it. What is the result? What metal is indicated?

¹ During the exercises upon the separation of sulphur from sulphides by nitric acid, the ventilation of the laboratory may demand some attention.

(b) Make a tube sealed at one end (Ex. 1, B). Place in it 0.5 c.c. of crushed pyrite and heat it in the flame of the burner, after removing the wing-top. What element do you detect? If in doubt, break the tube, scrape out the substance in the upper part with a little wad of asbestos, burn, and notice the odor.

(c) Treat 1 c.c. of powdered pyrite with 2 c.c. of nitric acid and carry out directions of part (c) of the preceding exercise with it. Avoid inhaling the gas given off when the nitric acid interacts with the mineral.

What element do you detect? What is the color of pyrite? What is the form of the crystals? Make a drawing of a crystal in your notebook. Is it hard or soft? Can you scratch it with a knife?

EXERCISE 8

CHALCOPYRITE

(*Copper Pyrite*)

Apparatus. — Blowpipe. Wing-top burner. Magnet. Funnel. Small test tube. Bottle.

Materials. — Chalcopyrite. Pyrite. Asbestos. Prismatic blow-pipe-charcoal. Iron nails. Glass tubing.

Method. — See textbook, page 14.

Examine chalcopyrite and compare it with pyrite, especially in color, hardness, and crystalline form.

(a) Heat a 2 mm. (0.08 in.) fragment on charcoal just as you did with pyrite. Test the residue with a magnet. What is the result?

(b) Carry out directions of part (b) of Exercise 7 with chalcopyrite. What element do you detect?

(c) Carry out directions of part (c) of the preceding exercise, using 1 c.c. of powdered chalcopyrite and 2 c.c. of concentrated nitric acid. Receive the liquid which passes through the asbestos in a bottle.

Burn the solid which remains on the asbestos. What is it?

Note the color of the liquid in the bottle. This color is characteristic of *copper* compounds dissolved in water. Dilute the liquid to at least 200 c.c., add four to five drops of concentrated sulphuric acid and place an iron nail or a piece of zinc in the liquid. Describe the substance which is deposited. What is it? What three elements do you detect in chalcopyrite?

EXERCISE 9

ARTIFICIAL SULPHUR-COMPOUNDS

Apparatus. — Small, dry test tube. Trip scales. Asbestos board. Lens. Medicine dropper. Mortar and pestle. Wing-top burner. Blowpipe. Prismatic charcoal.

Materials. — Flowers of sulphur. Iron wire. Zinc dust. Mercury. Thin sheet copper (0.05 mm. thick). Lead foil or test lead.

Method. — For copper sulphide, see textbook, page 18, section 23. For lead sulphide, see textbook, page 11. For zinc sulphide, see textbook, page 17. For mercury sulphide, see textbook, page 16.

(a) Cut a piece of sheet copper 4×12 cm. (1.6×5 in.). Weigh it on the trip scales. Fold it parallel to the long diameter so that the cross section forms a W (Fig. 9), and satisfy yourself that it will slip easily into a small, dry test tube. Make a hole near the edge of the copper so that you can hang it from an iron wire.

Fill the test tube 5 cm. (2 in.) deep with flowers of sulphur, clamp the tube near the mouth, and heat it until the sulphur boils violently and the tube is filled with the vapor.

Lower the copper into the sulphur vapor (not into the liquid). Does there seem to be any evolution of heat? When the action is complete, withdraw the product carefully and weigh it. Result? Is the product crystalline? (Use a lens and, if necessary, examine results obtained by other students.) Does the product resemble copper or sulphur in its properties? Proceed at once with (b).

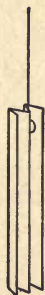


FIG. 9.

(b) Introduce into the same test tube a mass of powdered lead (test lead) about equal in volume to the sulphur, or use lead foil torn into small pieces, and take about six times the weight of the sulphur. If you do not know this, estimate it from the volume, remembering that 1 c.c. of liquid sulphur weighs 2 grams. Heat. Does the product resemble galenite? Does it behave like galenite when heated on charcoal before the blowpipe? Try it.

(c) Tear a sheet of paper in halves and place half of it on each side of the trip scales. The paper on the left-hand pan avoids contact between the substances weighed and the scales. The paper in the right-hand pan counterpoises that on the left. Use this method hereafter in weighing substances with the trip scales. Weigh off 2 grams of zinc dust and 1 gram of flowers of sulphur.

Mix the sulphur and the zinc by putting them together and pouring repeatedly from one paper to another. Transfer the mixture to the asbestos board and ignite it with the burner held at arm's length. The action is explosive. The product is a compound of sulphur and zinc which has the same composition as zinc-blende.

CAUTION: Remove all rings while working with mercury. Do not get mercury in the sinks.

(d) Melt 3 grams of sulphur in a small, dry test tube, add 1 c.c. of mercury with a medicine dropper, and continue heating. When the action is over, allow to cool. The mass consists of a compound of sulphur and mercury — called *mercuric sulphide* — which has the same composition as the mineral *cinnabar*. Powder the product in a mortar and examine it.

EXERCISE 10

SYNTHESIS OF COPPER SULPHIDE (CUPROUS)

(Quantitative)

Apparatus.—Porcelain crucible and cover. Balance. Pipe-stem triangle. Meter stick (one for section).

Materials.—Flowers of sulphur. Copper wire No. 30 (B. & S. gauge). A meter of this wire weighs about 0.5 gram.

Method.—When copper wire is heated with sulphur, copper sulphide, Cu_2S , is formed. Only the sulphur which combines with the copper remains in the crucible. The excess of sulphur burns off. See textbook, page 21.

Weigh the crucible without the cover. Coil 2 m. of the wire around your pencil into a spiral, place in the crucible, and weigh again. Count the weights at least three times to avoid errors.

Record thus:—

Crucible + copper
Crucible empty	<u>.</u>
Copper

Place about a gram (roughly weighed) of flowers of sulphur in the crucible, cover, and support on the triangle. Heat gently until sulphur no longer burns between crucible and cover; then heat intensely for five minutes. *Take the burner in the hand and make sure that the upper part of the crucible where it joins the cover is thoroughly heated.* Let cool and weigh, after removing cover. Do not remove cover until cold.

Record thus:—

Crucible + copper sulphide
Crucible + copper	<u>.</u>
Sulphur in copper sulphide
Crucible + copper sulphide
Crucible empty	<u>.</u>
Copper sulphide

Calculate the percentage composition of copper sulphide thus:—

$$\frac{\text{weight of copper} \times 100}{\text{weight copper sulphide}} = \text{per cent copper.}$$

$$\frac{\text{weight sulphur} \times 100}{\text{weight copper sulphide}} = \text{per cent sulphur.}$$

How do your results agree with the composition calculated from the formula Cu_2S ? See textbook, pages 89 and 385.

Calculate the atomic weight of copper, taking that of sulphur as 32. Since the formula of your sulphide is Cu_2S , two atomic weights of copper would combine with one of sulphur.

Hence:—

$$\begin{aligned} \text{Weight sulphur} : \text{weight copper} &:: 32 : 2x, \\ x &= \text{atomic weight copper.} \end{aligned}$$

In these calculations, “weight of sulphur” means the sulphur which *combines* with the copper; that is, the increase in weight after heating. The quantity of sulphur taken need not be accurately weighed, since the excess of sulphur is driven off as vapor and burns.

In this exercise, and in all quantitative work, the student should *use the utmost care to avoid errors in weighing*. Unless the weighings are correct the work is little better than a waste of time. *The weights should be counted at least three times, and the result recorded methodically at once.*

EXERCISE 11

THE CYANIDE PROCESS FOR EXTRACTING GOLD
FROM ITS ORES

Apparatus. — 300 c.c. flask with solid rubber stopper. Tinner's shears. Glass rod.

Materials. — Gold leaf. Potassium cyanide. Sheet zinc. (The zinc should be cleaned with emery paper before using.)

Method. — See textbook, page 29.

CAUTION: Potassium cyanide is intensely poisonous.

(a) Place 1 gram potassium cyanide with 50 c.c. water in the flask, insert the stopper, and dissolve by shaking. Introduce about 10 sq. cm. (1.5 sq. in.) of gold leaf. The gold leaf is best picked up with a wet glass rod, which is then dipped into the solution in the flask.

(b) Insert the stopper and shake. At intervals remove the stopper to admit oxygen, which takes part in the change. Result? What stage in the cyanide process does this illustrate?

(c) When the gold has disappeared, add 2 sq. cm. (0.3 sq. in.) of freshly scraped sheet zinc, cut into small pieces, and shake vigorously. Result? Remember that finely divided gold is dark and without luster. What part of the cyanide process does this illustrate?

EXERCISE 12

THE EFFECT OF HEAT ON WOOD AND SOFT COAL

Apparatus. — Dry test tubes. (Since the test tubes are to be sacrificed, damaged ones are preferable.)

Materials. — Sawdust or fragments of wood. Soft coal. Red and blue litmus paper.

Method. — See textbook, pages 41 and 43.

(a) Fill a dry test tube 2 cm. (0.8 in.) deep with sawdust or fragments of wood. Heat at first gently, then strongly. Test gases given off as to odor, effect on both kinds of litmus paper, and combustibility. What is left in the tube?

(b) Repeat (a), using soft coal instead of sawdust. Make a comparison of the results of (a) and (b) in your notes.

EXERCISE 13

WOOD CHARCOAL AND ANIMAL CHARCOAL
(BONEBLACK)

Apparatus. — Funnel. Small test tube.

Materials. — Wood charcoal. Animal charcoal. Litmus solution. Lead foil (tea lead). Filters.

Method. — See textbook, pages 41 and 194.

(a) Place a 1 cm. (0.4 in.) piece of wood charcoal in a test tube half full of water. Is it lighter or heavier than water? Now wrap the charcoal in lead foil to sink it and punch holes in the foil with a knife blade to admit the water. Put it back in the test tube and boil five minutes. Remove the foil and put the charcoal again in water. Result? Draw conclusions with respect to the true and apparent specific gravity of charcoal.

(b) Prepare a dilute solution of litmus by adding one or two drops of the laboratory solution to half a test tube of water. Add enough boneblack to fill the tube 2 cm. (0.8 in.) deep. Boil several minutes and filter. Fold the filter paper first into a

semicircle, then into a quadrant (Fig. 6), and fit it tightly to the funnel. Avoid filling the funnel above the filter.

What is the action of boneblack on litmus solution? What is boneblack used for?

EXERCISE 14

HEATING METALS IN THE AIR

Apparatus.—Pliers (one pair for class). Forceps. Porcelain crucible. Pipe-stem triangle. Platinum foil (one piece will answer for class). Tinner's shears. Meter stick. Balance.

Materials.—Sheet copper (0.1 to 0.2 mm. thick). No. 30 copper wire. Tin foil. Iron powder (by alcohol). Iron wire. Zinc dust. Sandpaper.

Method.—See textbook, page 49.

(a) Cut two pieces of copper about 10 cm. (2 in.) square. Clean both pieces with sandpaper. Was the copper tarnished? Is it likely, then, that the air acts upon copper? If so, the action must be very slow, but all chemical changes are greatly quickened by heat.

Hold one of the pieces of copper in forceps and heat it as hot as you can without melting it. When the product of heating begins to peel off, let the copper cool, place a clean paper on your desk, and tap the copper upon it so as to collect some of the new substance. Clean the copper with sandpaper and heat it again.

Would heat alone, without air, produce the change? In order to answer this question, roll up the second piece of copper, squeeze the roll flat with the pliers, and fold over the ends so that air will not get at the inside. Heat as hot as you can, without melting, for five minutes. Let cool, and compare the outside of the roll with the inside. Result?

(b)¹ Measure roughly 5 m. (5.5 yds.) of No. 30 copper wire, coil it around a lead pencil, and place in a porcelain crucible. Weigh accurately and record the weight. Place the crucible

¹ (b), (c), (d), (e), can be distributed to different students.

on a pipe-stem triangle and heat gently. Slowly increase the heat until you are using the full power of the burner. The crucible should be just above the tip of the inner blue cone of the flame.

After fifteen minutes, turn the flame to one half its full height for a minute (to prevent sudden cooling and cracking of the porcelain) and let the crucible cool. Weigh accurately. Result? What are your conclusions?

(c) Heat a piece of tin foil 5 cm. (2 in.) square as in (b). The *crucible with the tin foil* must be accurately weighed. Stir from time to time with an iron wire, being careful not to lose anything from the crucible. After at least fifteen minutes full heat, cool gradually and weigh again. Result?

(d) 2 c.c. of iron powder, accurately weighed with the crucible, are heated and stirred as in (c) and then reweighed.

(e) 2 c.c. of zinc dust heated exactly as in (c).

(f)¹ Take a piece of platinum foil in the forceps and heat it to bright redness for two minutes. Platinum belongs to the class of "noble metals." Let the platinum cool and notice the absence of alteration.

EXERCISE 15

THE COMBUSTION OF PHOSPHORUS IN AN ENCLOSED VOLUME OF AIR

Apparatus. — Bottle. Agate pan. Graduated cylinder. Small, flat cork. Glass plate 8 cm. square.

Materials. — Red phosphorus. Asbestos paper. Wooden splints. Pins.

Method. — Phosphorus combines with the *oxygen* of the air and does not affect the nitrogen.

Pin a piece of asbestos paper to the cork, place on it 1 c.c. of red phosphorus, and float it on water in the agate pan. Ignite the phosphorus and at once invert over it the bottle.

¹ For all students.

Wait until the white cloud of phosphorus pentoxide, P_2O_5 , has disappeared. Slip a glass plate tightly under the mouth of the bottle and remove it from the pan without allowing any air to enter. Set it upright upon the desk. Light a splint, remove the glass plate, and instantly plunge the burning wood into the gas. Pour the water into a graduated cylinder and measure its volume. Get the total volume of the bottle. Calculate the percentage of oxygen in the air.

How does the result compare with the accurate figure? (See textbook, page 57.) Should you expect an exact result or merely a rough approximation? What are the chief causes of error in the method? Should you expect the result to be low or high? Why?

EXERCISE 16

THE PERCENTAGE OF OXYGEN IN AIR

Apparatus is shown in the figure (No. 10). A large test tube is closed by a doubly perforated rubber stopper (No. 5). One hole is tightly plugged with a bit of thick glass rod, the ends of which have been rounded in the flame. Through the other hole passes a tightly fitting short piece of glass tubing which reaches 1 cm. (0.4 in.) below the stopper. The lower end of this tube has been held in the flame until it has melted sufficiently to reduce the bore about one half. A rubber tube 15 cm. (6 in.) long connects the upper end of this tube with a funnel as shown in the figure. The rubber tube is provided with a Mohr pinchcock.

Materials. — Dissolve 200 grams of potassium hydroxide in 1 liter of water. Add 20 grams of pyrogallol, which will dissolve instantly in the liquid. This solution will serve for at least 20 students. It should be kept in a well-stoppered bottle. Or, dissolve 10 grams potassium hydroxide in 50 c.c. water, add 1 gram pyrogallol, stir, and use at once. Rubber bands.

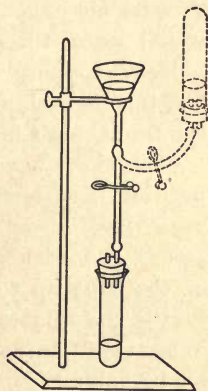


FIG. 10.

Method. — A solution of pyrogallol absorbs the *oxygen* from a measured volume of air, leaving the nitrogen.

CAUTION: *Potassium hydroxide must not be touched with the fingers nor weighed on the bare balance pan.*

Remove the test tube and take out the glass rod from the stopper. Pour about 40 c.c. of the solution just mentioned into the funnel. Open the pinchcock until the liquid has filled the rubber tube and the glass tube completely. If this is carefully done, scarcely any of the liquid need be allowed to run out. The solution stains the flesh and should not be allowed to touch the hands. It spoils on exposure to air, and the experiment should be finished as promptly as possible after the solution is put into the funnel.

Now insert the stopper tightly, and then plug up the second hole of the stopper with the piece of glass rod. Open the pinchcock. A little of the solution will be forced into the test tube by the pressure of the liquid above, and, as the oxygen disappears, more will enter. When the liquid stops flowing in, close the pinchcock and turn the test tube upside down several times to absorb the last traces of oxygen. In doing this, do not take hold of the tube with the hand, since this would warm the gas and give a false result. Use a paper holder or a clamp. Open the pinchcock and bring the inverted test tube to such a height that the level of the liquid in it and the funnel are the same. Why?

Close the pinchcock, bring the test tube back to its original position, and mark the lower level of the stopper and the upper level of the liquid with rubber bands. Clean the apparatus, and with a graduated cylinder measure the capacity of the tube up to the lower and upper rubber bands. The first is the volume of the oxygen, the second that of the air. What result do you obtain for the percentage of oxygen in the air by volume?

Calculate thus: —

$$\frac{\text{volume to lower band} \times 100}{\text{total volume to upper band}} = \text{per cent oxygen.}$$

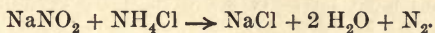
EXERCISE 17

CHEMICAL NITROGEN

Apparatus. — Small flask, with rubber stopper, thistle tube, and delivery tube for collecting over water (Fig. 11). Agate pan. Wire cutter. Three bottles. Wire gauze.

Materials. — Sodium nitrite (commercial crystals). Ammonium chloride (commercial). Iron wire No. 20. Christmas-tree candles.

Method. — Nitrogen escapes when a solution containing sodium nitrite and ammonium chloride is heated :—



Put about 10 grams of ammonium chloride and the same quantity of powdered sodium nitrite, NaNO_2 , in a small flask, and add 80 c.c. water. Set up the apparatus as shown in Fig. 11. Apply a gentle heat, let the gas escape for three minutes. Why? Collect three bottles of the nitrogen over water. *Stop heating as soon as the reaction begins, or it will become too violent.* If the evolution of gas threatens to become too energetic, immerse the generating flask *for an instant only* in a pan of cold water, which should be in readiness. This will immediately quiet it.

What are the physical properties of the substance you have prepared? Does it burn? Does it support the combustion of a candle? What is the most important difference between this nitrogen and that you have obtained from the air (Exercises 15 and 16)? This nitrogen is a little lighter than nitrogen obtained from air. Why?

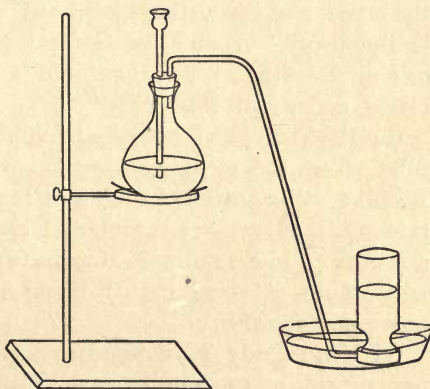


FIG. 11.

EXERCISE 18

OXYGEN

(Preliminary Experiments)

Apparatus.—Hard glass test tube. Watch glasses about 8 cm. diameter. Beaker. Small test tubes. Lens. Wire gauze.

Materials.—Potassium chlorate. Manganese dioxide. Mercuric oxide. Wooden splints.

Method.—For mercuric oxide, see textbook, page 53. Potassium chlorate, when heated, passes into oxygen and potassium chloride:—



The change takes place at a lower temperature in the presence of manganese dioxide.

(a) Shake up some potassium chlorate with cold water in a test tube. Pour off a few drops of the clear solution into a watch glass which serves as a cover for a beaker half full of hot water. The beaker is placed on wire gauze on a ring of the stand, and the water is kept hot by frequent heating to the boiling-point. When crystals form, examine them with a lens or a microscope. While the liquid in the watch glass is evaporating, go on with (b).

(b) Heat 2 c.c. of potassium chlorate in a dry, clean test tube. Test the escaping gas with a splint bearing a spark. Result? What is left in the tube can only be potassium chlorate minus oxygen. Let cool, dissolve in water, and evaporate a few drops to crystallization as in (a). Compare the crystals with those obtained in (a), using a lens. They consist of potassium chloride.

(c) Have ready 2 c.c. potassium chlorate and 2 c.c. manganese dioxide on separate papers. Place the chlorate in a small, dry test tube, clamp in a vertical position, and melt the chlorate by the cautious use of a small flame. Avoid overheating. Show by the spark that you have not applied a high enough temperature to drive off any oxygen.

Withdraw the flame. At once throw the manganese dioxide into the melted chlorate and test with the spark. Result? The manganese dioxide remains unaltered. What effect has it upon the change of the potassium chlorate to potassium chloride?

CAUTION: Rings should be removed from the hands before beginning (d), as mercury alloys with gold and turns it white. Do not get mercury into the sinks.

(d) Heat 2 c.c. mercuric oxide in a hard glass test tube. Use the spark test. Result? What remains in the tube?

EXERCISE 19¹

OXYGEN

(Preparation)

Apparatus.—Large test tube with rubber stopper and delivery tube. Four glass plates 8 cm. square. Agate pan. Iron spoon. Four wide-mouthed bottles. Beaker. Wire cutter.

Materials.—Iron wire. Christmas-tree candles. Picture cord. Potassium chlorate. Manganese dioxide. Sulphur. Red phosphorus. Sand. Asbestos paper.

Method.—See textbook, pages 54, 55, and 56.

(a) Mix on paper 20 c.c. potassium chlorate with 10 c.c. manganese dioxide. Place the mixture in the test tube. Hold the tube in a horizontal position and tap it gently so as to make a channel along the upper side of the mass for the escape of gas—otherwise explosions may occur. Clamp in a horizontal position and tightly insert stopper bearing delivery tube, leading to agate pan of water. Four inverted bottles filled with *water* (no air bubbles) should be ready in the pan.

Heat gently, beginning at the top. Obtain a steady evolution of gas. If the latter comes off violently, stop the heat until it moderates. Keep the burner in motion and *do not heat any portion of the tube hot enough to color the flame yellow.*

¹ Two students working together.

From time to time, take out water with a beaker from the pan so as to keep the latter about half full. Bottles full of oxygen can be covered *under water* with a glass plate and set in the desk *right side up* until used.

(b) In the following experiments, the bottles should be allowed to stand on the desk and the glass plate slipped aside just enough to allow the admission of the burning substance, which should be held near, but not touching, the bottom. The flame should not be allowed to come in contact with the bottle, as it will crack the glass.

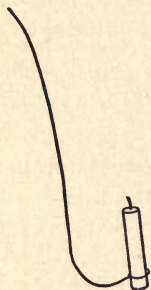


FIG. 12.

Fasten a candle in a wire as in Fig. 12, light and place in oxygen. Keep the flame uppermost. Result? Keep the bottle covered. How does the flame differ from that of a candle burning in air, and why? Why is it finally extinguished? Does the candle seem to burn more or less rapidly in oxygen than in air? If it burned at the same rate, how much longer would it burn in a closed bottle of oxygen than in the same bottle filled with air? How much greater is the *concentration* of oxygen in pure oxygen than in air under the same pressure? Follow the same line of thought in interpreting the other three combustions.

(c) Line a clean iron spoon with asbestos paper, fill it with red phosphorus, ignite, and place in oxygen as in (b). Does the product seem to be a solid or a gas? Name it. Clean the spoon for the next experiment by heating it to redness for two minutes. It should be held nearly vertical during this process, so that the melted phosphorus may not run out. Then let it cool completely and reline it for the next experiment.

(d) Fill the clean, cold spoon with sulphur, light, and place in oxygen as in (c). Result? Name the product. Does it seem to be a solid or a gas? Cautiously note the odor. Reserve the sulphur in the spoon for (e).

(e) Slip aside the cover of the fourth bottle of oxygen an instant and throw in 10 c.c. of sand. Replace the cover.

Carefully melt the sulphur left in the spoon from (d) and dip into it one end of a piece of iron picture cord 25 cm. (10 in.) long. Ignite the sulphur, and lower the picture cord into a bottle of oxygen. Result? What was the object of using the sand? Of using the sulphur? *Do not get the sand into the sinks.*

EXERCISE 20

CARBON DIOXIDE

(Preliminary Experiments)

Apparatus. — Deflagrating spoon. Trip scales. Glass tube about 35 cm. (14 in.) by about 7 mm. (0.3 in.) diameter. Agate pan. Glass plate. Three wide-mouthed bottles of about 400 c.c. capacity. Beaker.

Materials. — Limewater. Charcoal powder. Splints. Christmas-tree candles. Iron wire. Asbestos fiber. Alcohol. Kerosene.

Method. — See textbook, pages 98-104.

(a) **Production of carbon dioxide by combustion.** — Fill a deflagrating spoon with powdered charcoal, heat the charcoal to redness, and let it burn in a covered bottle. Test the gas with limewater.

Hold a dry clean bottle over a *small* gas flame for a few seconds. What product do you notice? Apply the limewater test. What is the second product? Repeat with a candle flame. With burning wood. With the flame of kerosene. Of alcohol. (The last two flames can be conveniently obtained by dipping a bunch of asbestos into the corresponding liquid, placing it on the base of your stand, and setting fire to it.)

Draw conclusions regarding the existence of carbon in combustibles and the products of their combustion. State the evidence.

(b) **Changes produced in air by respiration.** — Place some clean limewater in a beaker and blow gently through it by means

of a glass tube. Result? Invert three wide-mouthed bottles full of water in your agate pan and collect air in the first bottle from the beginning of an expiration. Withdraw the bottle from the water by means of a glass plate, slip the plate aside an instant and lower into the bottle a lighted candle. Does it burn as long as it would in the same volume of pure air? Why?

In the second bottle, collect air from the end of an expiration, using the *last portions* of air from the lungs. Test this with a candle. Result? The result is due partly to the small amount of oxygen, and partly to the large amount of carbon dioxide.

Fill the lungs with air and hold the breath as long as you can without discomfort. Collect the first of the expiration and test it with a candle. Does the result furnish any evidence of the diffusion of carbon dioxide upward, or of oxygen downward, in the lungs?

The body burns up about 220 grams of carbon in 24 hours, almost all of which is cast out through the lungs as carbon dioxide. Weigh off roughly this amount of charcoal on the trip scales in order to get an idea of the quantity. Of course the amount varies greatly in different people and in the same person at different times. The greater the activity and the lower the temperature of the surrounding air, the more active the internal combustion becomes.

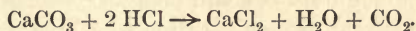
EXERCISE 21

PREPARATION OF CARBON DIOXIDE

Apparatus. — Gas-generating bottle. Iron spoon. Trip scales. Five wide-mouthed bottles. Small test tubes. Five glass plates.

Materials. — Cracked marble. Christmas-tree candles. Iron wire No. 20. Magnesium ribbon. Limewater. Litmus solution.

Method. — Calcium carbonate liberates carbon dioxide with acids (See textbook, page 321):—



Use the apparatus shown in Fig. 13. Place the gas bottle almost horizontal and slide enough broken marble into it to fill it when upright to the depth of 1 cm. (0.4 in.). If the lumps are too large, they can be broken with a hammer on an iron plate — not in a mortar. Add 50 c.c. of water, and then hydrochloric acid, slowly through the thistle tube until a brisk evolution of gas is obtained. Collect it by downward displacement in four dry bottles.

Investigate the physical properties of the gas. Has it any odor or taste? If in doubt about the last point, let the gas from the generator bubble through a little water in a test tube and taste the liquid. Test its solubility in water by pouring water 5 cm. deep into a bottle of the gas, covering tightly with the hand and shaking. Suction upon the hand indicates solution of the gas.

Lower a lighted candle into a bottle of the gas. In order to illustrate its high density, place a lighted candle on your desk and pour carbon dioxide over it, just as you would pour water. Balance a bottle on the platform scales and pour a bottle of the gas into it.

A few substances will burn in the gas. Try an 8 cm. (3 in.)

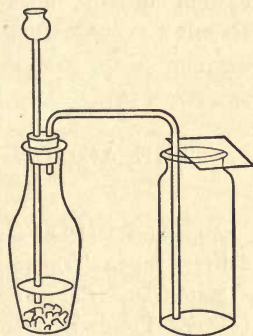


FIG. 13.

piece of magnesium ribbon wrapped round the stem of your spoon and start burning in the air. What is the black substance which is obtained along with the magnesium oxide?

Pass carbon dioxide through some limewater in a test tube for some time. Notice that the calcium carbonate, which is at first precipitated, finally redissolves. Calcium carbonate is soluble in water containing carbon dioxide, and this solution is present in many "hard waters."

Boil the liquid, and show that, when the carbon dioxide is expelled, the precipitate is again obtained

Add five drops of litmus solution to 10 c.c. of water in a test tube and pass carbon dioxide through the liquid. Result? The presence of the water is necessary to this change. Dry carbon dioxide does not affect litmus. Boil the liquid. Result?

EXERCISE 22

THE ACTION OF CARBON ON OXIDES OF METALS

Apparatus. — Hard glass test tube with one-hole rubber stopper and delivery tube. Trip scales. Beaker. Lens.

Materials. — Cupric oxide (CuO). Tin oxide (SnO_2). Lead oxide (PbO). Powdered charcoal. Limewater.

Method. — See textbook, page 142.

(a) Mix well on paper 5 grams cupric oxide with 0.5 gram powdered charcoal. Cut a piece of paper 20×1 cm. (8×0.4 in.) and crease it in the middle, making a V-shaped trough. Take up some of the mixture in the trough and, holding the latter in a horizontal position, slip the tube over it. Upset the trough and deposit the powder. Introduce the rest of the mixture in the same way, and use this method hereafter when it is necessary to get a powder into a tube without soiling the walls.

Clamp the tube in a horizontal position and insert the stopper, letting the delivery tube dip into 50 c.c. limewater in a

beaker (Fig. 14). Heat with a flame kept in constant motion. Watch the limewater closely and explain the changes which take place in it. When the action seems complete, remove the limewater *before* the burner. Let cool completely, take out the stopper, place the residue in the tube on paper and examine, using a lens if necessary.

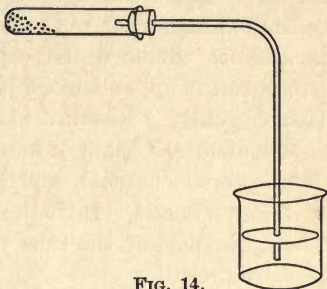


FIG. 14.

(b) Repeat, using tin oxide (5 grams) and charcoal (1 gram).

(c) Repeat, using lead oxide (2 grams) and charcoal (2 grams). Employ a gentle heat.

What is the practical importance of these results?

If the laboratory period is short, (a), (b) and (c) can be distributed and the students encouraged to compare results.

EXERCISE 23

ARSENOLITE (*Arsenious oxide*) AND ARSENIC

Apparatus.—Lens. Blowpipe. Evaporating dish. Small test tube.

Materials.—Arsenious oxide. Arsenic. Thin sheet copper. Prismatic charcoal. Powdered charcoal. Glass tubing. Filter paper.

Method.—See textbook, page 293, section 387. Also page 332.

(a) **Arsenious oxide.**—Examine the substance and compare it with the *element* arsenic. Heat a trace of arsenious oxide in a tube sealed at one end. Examine the sublimate with a lens. What is the shape of the crystals?

Dissolve about 0.1 gram of arsenious oxide by boiling it gently in a test tube with dilute hydrochloric acid. Dilute the liquid to 10 c.c.

Place a clean piece of sheet copper about 2 cm. square in a dish, fill the dish two thirds with water, add about 1 c.c. of

hydrochloric acid, and heat almost to boiling. Is there any action? Add 1 c.c. of your arsenic solution to the liquid, and continue heating for ten minutes. The deposit on the copper is *arsenic*. Remove the copper, dry it carefully with filter paper, roll it up, and place it in a glass tube sealed at one end. Heat it gently. Result? Look for a sublimate with a lens.

Mix about 0.1 gram of arsenious oxide with twice its weight of powdered charcoal, and heat the mixture in a glass tube sealed at one end. Introduce the mixture with a paper trough. The upper part of the tube must be *clean*. Describe and explain the result.

(b) **Arsenic.**—Heat a 1 mm. fragment of arsenic in a glass tube closed at one end. Does it melt? Does it vaporize? The two sublimates (steel-gray and black) are different allotropic forms of arsenic. Heat a fragment of arsenic the size of the head of a pin on charcoal with the blowpipe flame. The visible product is *arsenious oxide*. Note the odor of arsenic vapor.

EXERCISE 24

CARBON MONOXIDE¹

Apparatus.—Flask, dropping funnel, and delivery tube connected as shown in cut. Agate pan. Three bottles. Wire gauze. Three glass plates.

Materials.—Formic acid. Sulphuric acid (concentrated). Lime-water. Iron wire. Candles.

Method.—Formic acid, when heated with sulphuric acid, is converted into carbon monoxide and water. See textbook, page 203.

Set up the apparatus as shown in cut (Fig. 15). Clamp the flask firmly. The flask contains concentrated sulphuric acid

This method of making carbon monoxide is much safer and, in all respects, better than the one usually given (heating oxalic acid with sulphuric acid). Nevertheless it must be remembered that carbon monoxide is a *poisonous* gas.

to the depth of 1 cm. The dropping funnel is two thirds filled with formic acid. Warm the sulphuric acid gently with a

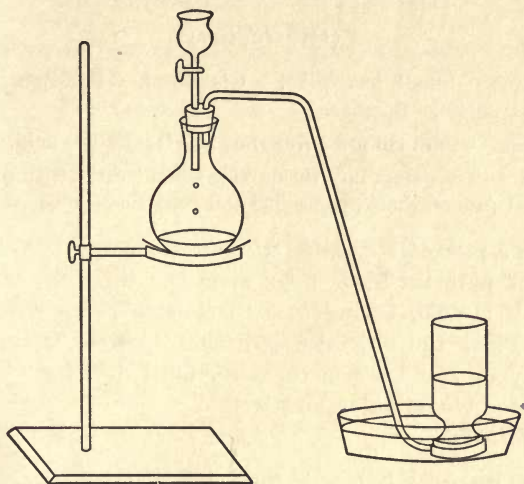
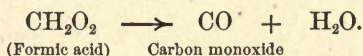


FIG 15.

small flame and allow the formic acid to drop slowly into it. The equation is: —



The water is retained by the sulphuric acid.

Make no attempt to ascertain the odor of the gas and do not allow it to escape unnecessarily. Turn off the stopcock when you wish to stop the evolution of gas.

Collect over water three bottles of the gas. Is the gas soluble in water? Does it burn? Does a candle burn in an inverted bottle of the gas? Shake up some limewater in a bottle of the gas. Result? Remove the cover an instant, ignite the gas, cover, and shake again. Result? What is the product of the combustion? Write the equation.

EXERCISE 25

THE ATOMIC WEIGHT OF TIN

(Quantitative)

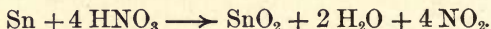
Apparatus.—Small test tube. Glass rod. Porcelain crucible. Pipestem triangle. Balance.

Materials.—Pure tin foil (*free from lead*¹). Nitric acid.

Method.—Tin passes into tin dioxide when treated with nitric acid and heated (see textbook, pages 298 and 299).

Weigh a porcelain crucible without the cover. Weigh in it 1 gram of pure tin foil. Pour upon the tin 2 c.c. of concentrated nitric acid, taking care to moisten every part of the metal. Place the crucible upon the pipestem triangle on a ring of your stand, warm carefully until dry, heat to bright redness for ten minutes and weigh.

Calculation:—



The product in the crucible is *tin dioxide*, SnO_2 , and the gain in weight is *oxygen*. Hence the atomic weight of tin is found from the proportion:—

Gain in weight : weight of tin :: 32 : atomic weight of tin.

¹ Supply houses have a strong tendency to furnish an alloy of tin and lead when pure tin foil is ordered.

EXERCISE 26

THE DISTILLATION OF WATER

Apparatus. — Flask, with rubber stopper and delivery tube bent as indicated in Figure 16. Bottle. Small test tube. Beaker. Glass rod (thin). Wire gauze.

Materials. — Table salt. Ammonia water. Red litmus paper. Potassium permanganate.

Method. — See textbook, page 112.

(a) Dissolve 3 c.c. salt in 50 c.c. water in a beaker by stirring. Place the liquid in the flask and arrange as shown in the figure. Boil gently until 3 to 5 c.c. have collected in the small test tube. Taste the distilled water. Is it free from salt? Explain. Repeat, without tasting, with 50 c.c. of water colored with a crystal of potassium permanganate.

(b) In a beaker, add 3 c.c. ammonia water to 50 c.c. water. Note the odor of the liquid and place a drop of it on red litmus paper.

After cleaning the apparatus, distill the dilute ammonia in the same way as you did the salt water. Note the odor of the distilled liquid and place a drop of it on red litmus paper. Is it free from ammonia? Explain.

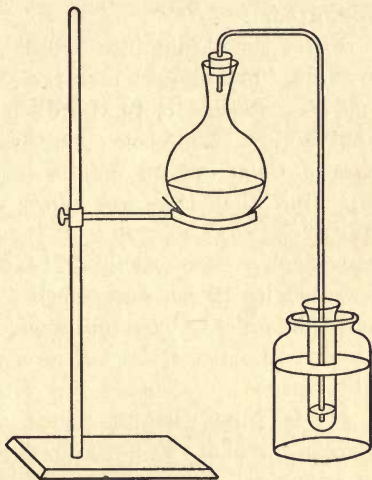


FIG. 16.

EXERCISE 27¹

CRYSTALLIZATION

Apparatus. — Wire gauze. Evaporating dish. Beaker. Graduated cylinder. Mortar and pestle. Funnel. Test tubes. Trip scales.

Materials. — Filters. Paper. Saltpeter (potassium nitrate). Bluestone (copper sulphate).

Method. — See textbook, page 207.

(a) Measure 50 c.c. of water into a dish. Make sure that the dish is dry on the outside, place it on wire gauze, and heat it to boiling. Gradually add 25 grams of potassium nitrate, stirring constantly with a glass rod. Heat until dissolved.

Filter the liquid into a beaker, wash the dish, and dry the outside. The liquid which runs through the filter is called the *filtrate*. Return it to the dish and boil away about half of the liquid. Let cool. Collect the crystals on a filter and spread them out on manila paper to dry. Is there reason to think that they are purer than the original material? Why?

In each of two test tubes, heat gently 5 grams of potassium nitrate with 10 c.c. water until dissolved. Do not boil. Cool one tube quickly by running water over it. Let the other cool slowly. Compare the two crops of crystals and explain the difference.

(b) If time permits, repeat the whole experiment, using *bluestone*, which you have powdered in a clean mortar, instead of saltpeter.

¹ If the laboratory period is short, (a) and (b) can be assigned to different groups of students.

EXERCISE 28**PREPARATION OF CRYSTALLIZED SODIUM CARBONATE**

Apparatus. — Evaporating dish. Graduated cylinder. Trip scales. Glass rod.

Material. — Anhydrous sodium carbonate (Na_2CO_3). Commercial calcined soda answers every purpose.

Heat 25 grams calcined soda with 60 c.c. water in a dish, stirring gently. If the liquid is not clear, filter it. Let cool. The crystals which form are the washing soda of the household ($\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$). Compare them with calcined soda. How could you reconvert them into calcined soda?

EXERCISE 29¹**SOLUTION (1)**

Apparatus. — (For each group.) Two beakers (100 c.c.). Two watch glasses suitable for covering them. Four small test tubes. Two corks to fit small test tubes. Wide-mouthed bottle. Graduated cylinder. Mortar and pestle. Two pieces wire gauze.

Materials. — Distilled water. Powdered rosin. Copper sulphate. Alcohol.

Method. — See textbook, pages 207, 208 and 214.

(a) One member of the group should use distilled and the other ordinary water. Carefully compare the watch glasses.

Clean a watch glass and put a few drops of distilled water in it. Use the watch glass as a cover for a beaker half full of water. Boil the water in the beaker gently until the water in the watch glass has evaporated. Meanwhile, go on with (b) and (c). If necessary, add more water to the beaker. If allowed to run dry, it will break. Examine the watch glass and set it aside.

¹ Two students working together.

With a second watch glass, test ordinary water in the same way, using about the same volume.

(b)¹ Place 1 c.c. of powdered rosin in a test tube, cover with water, and shake. Result?

Pour off the water, cover the rosin with alcohol, and shake. Result? Keep the liquid.

Shake up some copper sulphate powder first with alcohol and then with water. Explain the meaning of the terms *soluble*, *insoluble*, and *solution*.

Pour the alcoholic solution of rosin into a bottle nearly full of water. The resulting state of things is called a suspension. State, in parallel columns, some differences between solutions and suspensions.

(c) Since a liquid can only act at the surface of a solid, the more surface exposed, the more rapidly the solid dissolves. Therefore, when a solution is to be prepared quickly, the solid should be powdered.

Select two crystals of bluestone about 1 cm. (0.4 in.) in diameter and nearly equal in size. Have ready two small test tubes with corks to fit them. Powder one crystal finely in a clean mortar and introduce the powder into one of the test tubes. Place the unpowdered crystal in the other test tube. Add, from the graduated cylinder, 20 c.c. of water to each tube. Take the time with a watch and record it. Insert the corks and shake both tubes gently until the powdered bluestone is completely dissolved. Observe the condition of the crystal and estimate what fraction of it remains. Explain.

¹ If the laboratory period is short, one member of the group can perform (b) while the other carries out (c).

EXERCISE 30¹

SOLUTION (2)

Apparatus (for each group). — Two beakers (100 c.c.). Two watch glasses suitable for covering them. Two small test tubes with corks. Large test tube. Thermometer reading to 150° C. Graduated cylinder (100 c.c.). Trip scales. Two pieces wire gauze. Funnel.

Materials. — Calcium sulphate (powder). Chalk (powder). Thread. Calcium chloride (dry commercial). Sal ammoniac (commercial). Potassium carbonate (commercial). Ether. Carbon disulphide. Sodium thiosulphate (commercial “hypo”). Distilled water. Filters.

Method. — See textbook, page 214. Also page 302, section 400.

(a) One member of the group may take calcium sulphate; the other, chalk. Compare the watch glasses.

Place 1 c.c. of powdered calcium sulphate in a small test tube, add 10 c.c. distilled water, cork, and shake several minutes. Let settle, filter some of the liquid into a second clean test tube, and test it for dissolved matter as in Exercise 29, (a).

Treat chalk in the same way. Which is the more soluble? If you had neglected to filter and evaporate, what conclusion would you have formed about the solubility of both substances? Chalk is commonly spoken of as an “insoluble” substance. Is there anything *entirely* insoluble in water?

(b) Clamp a test tube containing 10 c.c. of water in a vertical position. From a ring of the stand hang a thermometer by a piece of thread so that the bulb is in the water. Weigh off 5 grams of dry calcium chloride on paper.

Boil the water with a small flame and take the temperature. Add the calcium chloride and when the solid is all dissolved take the boiling point again. Result?

(c) One member of the group may work with sal ammoniac, and the other with potassium carbonate. Carefully compare results.

Weigh 25 grams of sal ammoniac on the trip scales. Taking the specific gravity as 1.5, calculate the volume of the 25

¹ Two students working together.

grams. Measure 80 c.c. of water in the graduated cylinder, take the temperature of the water, and add the sal ammoniac. Cover the cylinder tightly with the band and invert repeatedly until the sal ammoniac is all dissolved. Take the temperature of the solution and read the volume.

Is the volume of the solution equal to the sum of the volumes of the substances it contains? What kind of a heat effect occurs?

Repeat with 25 grams of *potassium carbonate* (commercial pearlash). In the calculation, take the specific gravity of potassium carbonate as 2.

(d) One student may use ether; the other, carbon disulphide.

Liquids may dissolve each other.

CAUTION: Ether and carbon disulphide must not be used near a flame. Employ no heat in these experiments.

Place 3 c.c. carbon disulphide in a dry, large test tube. Add 3 c.c. water, one drop at a time, shaking constantly. Does carbon disulphide dissolve water perceptibly?

Now fill the tube to within 5 cm. of the top with water, cork and shake. Does water dissolve carbon disulphide to any large extent?

Repeat, using 3 c.c. of ether. Is water soluble in ether? Is ether soluble in water?

(e) **Supersaturated solutions.** — Place a few drops of water in a test tube, half fill the tube with sodium thiosulphate, — called “hypo” by the photographer, — and heat with a small flame kept in constant motion. When complete solution has occurred, pour the liquid into a clean tube and cover it with paper to exclude dust. Let it cool. No solid should separate. Throw into the cold liquid a crystal of solid sodium thiosulphate. Result? What is a *supersaturated* solution? Do you regard it as a stable or an unstable state of things? Would *any* crystal answer the purpose, if dropped into the supersaturated solution in this experiment? See textbook, pages 209 and 215.

EXERCISE 31

WATER OF CRYSTALLIZATION

Apparatus. — Eight clean, dry, small test tubes. Evaporating dish.

Materials. — Crystals of sodium sulphate. Copper sulphate. Zinc sulphate. Potassium sulphate. Calcium sulphate (cleavage pieces of gypsum). Alum. Barium chloride. Potassium nitrate. Alcohol.

Method. — Water of crystallization is driven off as steam when crystals containing it are gently heated.

Gently heat a few crystals of each substance in a dry, clean test tube. Use a separate tube for each experiment and clamp it in a horizontal position, so that the water may not run back into the hot portion and break it. The flame should be kept in constant motion, and the heat should not be intense enough to color the flame yellow.

Classify these eight substances into (1) those which contain water of crystallization and (2) those which form *anhydrous* (water-free) crystals. What change in structure occurs when the water is driven off from the first set? Note that anhydrous crystals may contain *traces* of water as an impurity.

Attend especially to the striking change in the case of copper sulphate. Dissolve most of the heated copper sulphate (it is anhydrous copper sulphate, CuSO_4) in the smallest possible quantity of hot water, transfer the liquid to a dish and let it cool. Explain. Throw the rest of the anhydrous copper sulphate into 5 c.c. of 95 per cent alcohol. What is the result? Remember that the remaining 5 per cent of the alcohol is chiefly water. Suggest a test for water based upon this behavior.

EXERCISE 32

WATER OF CRYSTALLIZATION

(Quantitative)

Apparatus.—Small porcelain crucible without cover. Pipestem triangle. Balance.

Materials.—Barium chloride. Magnesium sulphate. Copper sulphate. Calcium sulphate (coarsely powdered gypsum).

Method.—The water is driven off by heat and its quantity obtained from the loss in weight.

It is convenient to assign the different substances to different groups of students.

Clean and dry the crucible and weigh it accurately. Weigh in it exactly one gram of barium chloride. Count the weights *at least three times* to avoid errors.

Record the weights at once, thus:—

Weight of crucible + barium chloride
Weight of crucible, empty
Weight of barium chloride taken	<u>.</u>

Adjust a ring of your stand to the proper height and support the crucible on it by means of a pipestem triangle. Heat, at first gently. After five minutes allow the bottom of the crucible to come to a *faint* red heat (*no hotter*) and maintain it at that temperature for ten minutes. Let cool and weigh when cool enough to handle.

Record thus:—

Weight of crucible + barium chloride
Weight of crucible + barium chloride (after heating)	<u>.</u>
Loss in weight (water)

$$\frac{\text{loss in weight} \times 100}{\text{weight of barium chloride taken}} = \text{per cent of water.}$$

The other three substances are treated in the same way except that the temperature required is different.

Copper sulphate should be heated ten minutes with a half-sized flame kept in motion. No part of the crucible should glow. After weighing, the product can be dissolved in the crucible in the smallest possible quantity of hot water and allowed to cool. Result?

Calcium sulphate should be heated gently for five minutes. It should then receive the highest temperature the burner will yield for ten minutes, after which it should be cooled *gradually* by lowering the flame to half size for a minute before turning it out, *otherwise the crucible may crack*. Crucibles which have been highly heated should *always be cooled in this way*.

Magnesium sulphate should be heated in the same way as calcium sulphate.

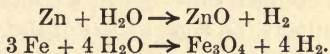
EXERCISE 33¹

THE ACTION OF ZINC AND IRON ON WATER

Apparatus. — Small test tube with rubber stopper and delivery tube. Medicine dropper. Glass plate. Bottle. Agate pan.

Materials. — Zinc dust. Iron filings. Wooden splints.

Method. — Heated zinc dust or iron powder liberates hydrogen from steam.



(a) Hold the tube vertical and let fall with the medicine dropper 10 drops of water to the bottom. Do not wet the sides. Cut a slip of paper 20×1 cm. (8×0.4 in.) and fold it into a little V-shaped trough which will slip into the tube. Holding the tube in an inclined position, put in with the paper trough enough zinc dust to soak up the water completely. Hold the tube nearly horizontal and deposit twice the quantity of *dry* zinc dust at *D* just above the wet zinc dust. Clamp the tube in a horizontal position and insert the stopper with the delivery tube leading to a pan of water (Fig. 17).

¹ (a) and (b) can be distributed to different students.

Heat *D*, gently at first, then as intensely as you can without softening the tube, then brush *W* with the flame so as to cause steam to pass over the hot zinc. Return constantly to *D* with the flame to keep it heated. Allow the gas to escape for the first minute. Why? Then collect it in an inverted bottle full of water. When the bottle is full, slip a glass plate under it

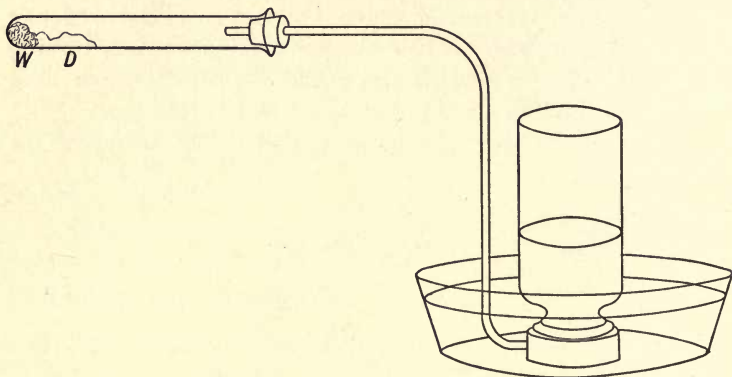


FIG. 17.

and remove it from the water. Keeping the bottle inverted, thrust a burning splint up into the gas. Is the gas combustible? Does it support the combustion of the wood?

Assuming that the white substance formed in the test tube is a compound of zinc and oxygen, what conclusion can you draw regarding the composition of water? If this explanation is correct, what action should we expect when iron is heated with steam?

(b) Clean the test tube and repeat the experiment, using iron instead of zinc.

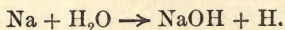
EXERCISE 34

THE INTERACTION OF SODIUM AND WATER

Apparatus.—Wide-mouthed bottle. Beaker. Watch glass. Wire gauze. Small test tube. Agate pan.

Materials.—Sodium. Thin lead foil (tea lead). Red and blue litmus paper. Phenol phthalein solution.

Method.—Sodium liberates hydrogen from water:—



Sodium must not be touched with fingers which are in the least moist either with water or perspiration. Your desk must be dry when working with it, and everything with which you touch it must be scrupulously dry. The air acts rapidly upon sodium and it must not be exposed. Take only a small piece from the bottle at a time, and immediately cork the latter. The liquid over the sodium in the bottle is naphtha or kerosene, and the bottle must not be opened in the vicinity of a flame. No sodium must be put away under the desk nor allowed to remain lying about, since it may catch fire. None must be thrown into the waste jar, since it may ignite the paper or other substances which the jar contains. Return any unused portions to the bottle, or place in a vessel specially provided for that purpose.

Throw a clean piece of sodium, free from crust, 2 mm. in diameter—no larger—into a bottle one fourth full of water. Immediately cover the bottle with a piece of paper, and *wait until the reaction is over before removing the cover.* The action usually ends with a slight explosion which may endanger the eyes if the cover is removed too soon.

Describe what happens. Throw in another similar piece of sodium. Feel the water in the bottle between the fingers. Result? Taste a little of it. Result? *Here, and always, immediately reject the liquid tasted and rinse out the mouth with water. Do not taste substances unless directed.* Evaporate a few drops of the liquid to dryness in a watch glass which serves as a cover for a beaker half full of gently boiling water. Result? Try the behavior of the liquid with red and blue

litmus paper, pieces about 1 cm. (0.4 in.) square or less. Result? Allow to fall into the liquid a drop of a solution of phenol phthalein. Result? To what are these new properties of the liquid due?

Wrap a clean piece of sodium 2 mm. (0.08 in.) in diameter in dry lead foil (tea lead). Punch several holes in the lead with a knife blade. Invert a test tube full of water in an agate pan containing water, and quickly slip the lead containing the sodium under it. If necessary, use another smaller piece of sodium wrapped in lead to complete the filling of the tube. *Take no more sodium than is directed.* The use of larger quantities is likely to cause explosions which may imperil the sight.

What gas collects in the tube? Is it soluble in water? Does it burn? To what product? Give the evidence for the belief that the gas comes from the water, not from the sodium.

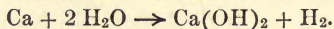
EXERCISE 35¹

THE INTERACTION OF CALCIUM AND WATER

Apparatus.—25 cm. (10 in.) glass tube open at both ends. Agate pan. Beaker. Small test tube.

Materials.—Calcium. Red and blue litmus paper. Phenol phthalein solution.

Method.—Calcium liberates hydrogen from water:—



Throw a 2 mm. (0.08 in.) bit of calcium into 50 c.c. of water in a beaker. Does gas escape?

The liquid is *limewater*, made in an unusual way. How does it behave with red and with blue litmus paper? Mix a few drops with a drop or two of phenol phthalein solution in a clean test tube. Result? Blow through the remainder of the limewater with the glass tube. Result?

Invert a test tube filled with water in the agate pan and

¹ Alternative to Exercise 34.

place under the mouth of the tube a bit of calcium. What gas collects? Is it combustible?

NOTE.—In spite of the most careful supervision, accidents occur during laboratory work with sodium. The interaction of *calcium* with water is perfectly safe and is in all respects a better experiment. Calcium can now be purchased quite cheaply, though some little inquiry may be necessary. The best way to subdivide it is to fasten in a vise and chip it up with a chisel. Manual Training Schools equipped with a “shaper” can cut calcium into shavings which answer perfectly in the laboratory. Keep in tight-stoppered bottle.

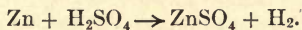
EXERCISE 36

HYDROGEN

Apparatus.—Gas-generating bottle with thistle tube and delivery tube for collecting over water. Agate pan. Four wide-mouthed bottles of about 400 c.c. capacity. Watch glass. Beaker. Small test tubes. Glass tubing. Wire gauze. Trip scales. Four glass plates.

Materials.—Mossy zinc. Wooden splints. Christmas-tree candles. Iron wire. Small rubber bands. Concentrated sulphuric acid. Copper sulphate solution. Copper oxide (black). Filters.

Method.—Zinc liberates hydrogen from dilute sulphuric acid:—



(a)¹ Place about 20 grams of mossy zinc in a gas-generating bottle. The bottle must be held almost horizontal and the zinc allowed to slide into it, otherwise the shock of the falling zinc will break it. The bottle is provided with a doubly perforated stopper carrying a thistle tube and a delivery tube, and the apparatus is arranged for collecting the gas over water.

Insert the stopper tightly with a twisting motion, and pour in through the funnel tube enough water to cover the zinc. The funnel tube must dip into this water. Extinguish any burner flame that may be in the neighborhood, and slowly add

¹ If the laboratory period is short, it will be necessary to omit or to postpone (b) and (c).

concentrated sulphuric acid through the funnel tube until gas is briskly evolved. Do not add too much acid. The maximum should be about one fourth as much by volume as there is water present. Allow the gas to escape through the water for three minutes. Why? *Do not attempt at any time in the experiment to light the gas at the exit tube.* If the gas does not come off freely, add a few drops of copper sulphate solution through the funnel tube.

Collect the gas over water in wide-mouthed bottles of about 400 c.c. capacity. Determine its properties. Has it any color or odor? Is it soluble in water? Does the method of collecting it throw any light on this last question? In order to obtain more definite information fill a test tube half full of hydrogen and mark the level of the water by a rubber band. Then shake the tube for a time, keeping its mouth under water. Result? Use this method hereafter in testing the solubility of gases.

Will the gas support combustion? Try it by holding a bottleful mouth downward and introducing a lighted candle fastened on a wire. Keep the candle out of contact with the walls of the cylinder so as not to wet the wick. Withdraw the candle slowly. Result? Repeat. *In all work with candles, support the candle on a wire so that the flame is uppermost.*

Fill two bottles of the same size with the gas. Support one in an inverted position in a ring of your stand, the mouth not touching the table. Place the other upright. Uncover them at the same moment and allow both to remain uncovered for one minute. Now thrust a lighted splint into each in turn. Draw conclusions.

Fill a test tube over water $\frac{5}{7}$ with air and $\frac{2}{7}$ with hydrogen. Ignite the mixture. Explain the cause of this behavior. Why does not the flame strike back from gas jets along the mains to the gas works? Would it be safe to supply cities with a *mixture of gas and air* by means of pipes?

(b) Take off the delivery tube from your gas bottle and substitute a straight tube which runs to the bottom of a dry test

tube clamped in a horizontal position. In this test tube is 1 c.c. of copper oxide powder. If necessary, add more acid through the funnel tube. *Wait at least three minutes for the air to be expelled*, then heat the copper oxide. Note the change in the copper oxide. Look in the cooler part of the tube for the other product. Explain. What other element most resembles hydrogen in its action on oxides?

(c) Filter a few drops of the liquid which remains in the gas bottle into a watch glass. Place it on a beaker half full of water, support the beaker on wire gauze, and boil the water gently.

Describe in your notes the crystals which separate. They are called *zinc sulphate*, and they contain zinc, sulphur, and oxygen. Sulphuric acid contains hydrogen, sulphur, and oxygen, so that we may describe the change which takes place in the gas-generating bottle by the statement that the zinc takes the place of the hydrogen.

EXERCISE 37

FLAME

Apparatus. — Platinum wire. Wire gauze. Sheet copper 20 cm. \times 15 cm. (8 \times 6 in.). Safety lamp.

Materials. — Wooden splints. Pins. Glass tubing. Glass rod. Charcoal. Candle. Touch paper.¹ Fine emery paper. Solution of one part potassium dichromate in 20 parts water, to which has been added one part sulphuric acid. Crayon.

Method. — See textbook, pages 122, 123, and 124.

(a) Take the chimney off the Bunsen burner. Light the gas at the tip in the base. Extinguish, replace the chimney, close the air holes, and light the gas. Do the size and shape of the orifice from which the gas escapes affect the flame? Formerly, when the naked gas flame was much used for lighting, the determination of the area and shape of the orifice which gave the best results was an important matter.

¹ Dip strips of filter paper 1 cm. wide in a cold saturated solution of potassium nitrate. Hang up to dry over a piece of glass tubing.

Turn the luminous flame down to a height of 2 cm. (0.8 in.) and hold a piece of crayon in it for a few seconds. Result? Open the air holes and hold a fresh piece of crayon in the blue flame for the same length of time. Result?

Turn the blue flame to full height. Stand with your back to the window and look through the flame at a dark background, *e.g.* the cover of a black book. Make a sectional drawing of the flame as it appears to you, indicating the different regions. Do the same with a candle flame.

The hottest part of the blue flame of the burner is level with the tip of the inner blue cone. Verify this by moving a platinum wire around in different regions, and noting where it glows most brightly. Note also that it is possible to obtain light from the blue flame by heating a solid body in it. This fact has revolutionized the methods of lighting by gas.

Hold a piece of glass rod about 3 mm. (0.1 in.) in diameter horizontally in the hottest portion of the blue flame until it sags perceptibly. Note the time required in seconds to produce this effect. Let the rod cool, and repeat with the luminous flame. Use a different portion of the rod, and hold it at about the same level in the flame as with the blue. Which flame appears to be more satisfactory as a source of heat?

Rub together two pieces of charcoal 1 cm. (0.4 in.) from the open air holes at the base of the burner giving the blue flame. Result? Does this confirm your experiment with the crayon? Judging from this, what is the direction of motion through the air holes? Does gas escape or air enter? Investigate this further by holding a piece of smouldering touch paper 1 cm. from the air holes. A splint which has just been blown out can be used almost as well.

(b) Polish a flat piece of sheet copper 20×15 cm. (8×6 in.) with fine emery paper. Hold it vertically in the center of the flame with one edge resting on the burner. The time required is only a few seconds. Judge by the eye when to remove it.

Thrust another portion of the copper horizontally through the hottest portion of the flame for a few seconds. Examine

the two sections of the flame obtained. What is the chemical nature of the discoloration on the copper? What difference does the experiment indicate regarding the chemical effects which might be expected in different parts of the flame? Wet the discolored parts of the copper with a glass rod which has been dipped into a solution of potassium dichromate containing sulphuric acid. Wash the copper and dry it. Card-board or stiff paper can be used instead of copper, but some skill is required to prevent ignition.

Hold in the center of the blue flame, 1 cm. above the bottom, a glass tube open at both ends inclined at an angle of 45° upward. Bring the flame of a splint to the upper end. Result? Blow out a candle and instantly apply a flame to the little column of smoke. Result?

Thrust a wooden splint horizontally through the blue flame of the burner 1 cm. above the base. Withdraw before it takes fire, and examine. Result? Support a match by a pin as indicated in Fig. 18 and light the blue flame.



FIG. 18.

(c) Hold a piece of wire gauze horizontal and bring it down vertically upon the blue flame. The gauze should be large enough to prevent the flame slipping around the edge. Result? Extinguish the flame, turn on the gas, and light it above the gauze. Result?

If a miner's safety lamp is available, examine it and record in your notes the peculiarity of its construction. Make a sectional drawing of it.

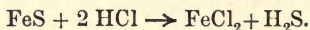
EXERCISE 38

HYDROGEN SULPHIDE

Apparatus. — Large test tube with rubber stopper and delivery tube bent twice at right angles. Large test tube. Three small test tubes.

Materials. — Iron sulphide in small fragments. Red and blue litmus paper. Solutions of mercuric chloride, copper sulphate and arsenious oxide. Sheet copper.

Method. — Many metallic sulphides yield hydrogen sulphide with hydrochloric or dilute sulphuric acid. (See textbook, pages 167, 168, 169):—



CAUTION: Hydrogen sulphide is poisonous. Avoid inhaling it.

Place the large test tube almost horizontal and carefully slide in enough iron sulphide to fill it 1 cm. (0.4 in.) deep. Add water to the depth of 3 cm. (1.2 in.) and then hydrochloric acid slowly until gas is briskly but not violently evolved. Clamp the tube upright and insert the stopper bearing a delivery tube. Collect a dry test tube of the gas by downward dry displacement. Light the gas in the test tube, after removing the delivery tube.

What are the two products of combustion? What evidence have you of the formation of each? What conclusion can you draw with respect to the composition of the gas?

Hold a piece of clean sheet copper close to the exit tube of your apparatus. A silver coin. Result?

Nearly fill a large test tube with water and let the gas escape through it for several minutes, running the delivery tube to the bottom. Stop the production of gas by filling the generating tube with water and pouring away the liquid. Use separate small portions of the solution of hydrogen sulphide for the following experiments. In each case, place the hydrogen sulphide solution in a test tube and add a few drops of a solution of the substances mentioned.

(a) Copper sulphate.

(b) Mercuric chloride.

(c) Arsenious oxide.

In (c) after noting the result, add a few drops of hydrochloric acid. Result?

Taste a little of the *original* hydrogen sulphide solution. Investigate its action on both kinds of litmus paper.

EXERCISE 39

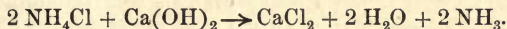
AMMONIA

Apparatus. — Large test tube, with stopper and delivery tube bent once at a right angle. Perforated square of cardboard. Two glass plates. Evaporating dish. Three small test tubes. Large test tube. Two bottles. Glass rod.

Materials. — Glue. Ammonium chloride. Ammonium sulphate. Dry slaked lime. Red litmus paper.

Method. — See textbook, pages 170, 172, 173.

A mixture of ammonium chloride and slaked lime yields ammonia:—



(a) **Formation from organic matter.** — Mix 1 c.c. of glue, powdered if necessary, with twice its volume of dry slaked lime. Heat the mixture *gently* in a dry, small test tube. Do not scorch the glue. Note the odor of the gas given off and its effect upon red litmus paper. *After testing* with litmus hold a glass rod wet with hydrochloric acid in the gas. Almost any organic matter containing nitrogen will yield ammonia if treated in this way. This can serve, therefore, as a *test* for nitrogen in such substances.

(b) **Formation from ammonium salts.** — Take 1 c.c. of ammonium chloride in one hand and an equal volume of slaked lime in the other. Notice the odor of each. Rub together. Note odor and apply the same tests as in (a).

Repeat with ammonium sulphate and slaked lime.

(c) **Preparation.** — Place enough ammonium chloride on a piece of paper to fill your generating test tube 5 cm. (2 in.)

deep. Add an equal volume of slaked lime. Mix well. Transfer to the test tube. Support the latter in a horizontal position as indicated in Fig. 19. The delivery tube passes upward through a perforated card or piece of pasteboard held by a ring on the upper part of the stand. The tube should reach to the bottom of the inverted bottle. Warm *gently*. Use a *small* flame. Avoid heating the *wet portion* of the tube, or breakage will re-

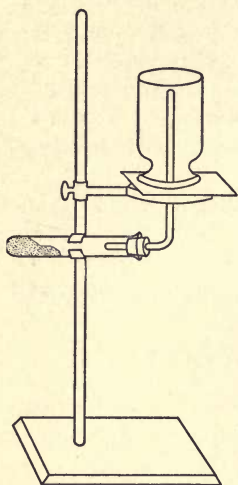


FIG. 19.

sult. Collect a dry bottle and a dry test tube full of the gas. Test when the bottle is full by holding a rod wet with hydrochloric acid just below the perforation in the cardboard. Slight fumes can be disregarded. A dense, white smoke indicates that the bottle is full and ammonia is escaping. Place the bottle mouth downward upon a glass plate until you are ready to use it. Keep the generator for (d).

Place your test tube of ammonia with the mouth under water in a dish. Shake gently. Result? Warm a clean, dry bottle by passing it rapidly through the burner flame. *Holding it in the flame will crack it at once.* Place in the bottle five drops of concentrated

hydrochloric acid and run the acid around the walls as much as possible, holding the bottle upright and keeping it covered with a glass plate. Now bring it mouth to mouth with the bottle of ammonia and remove both plates, keeping the bottles in contact. Invert the bottles several times, keeping them mouth to mouth. What is the deposit? Write the equation.

(d) Turn the delivery tube downward and bring it *just in contact* with the surface of 5 c.c. of water in a small test tube. Pass in the gas for five minutes. What is the action of the liquid on red litmus paper? What hydroxide must it contain?

NEUTRALIZATION OF AMMONIUM HYDROXIDE 53

Show by the tests of (a) that this hydroxide is decomposed and the ammonia driven out, by heating the liquid.

Place 5 c.c. strong ammonia water from your stock bottle in a small test tube and clamp in an inclined position with the clamp about at the middle of the tube. Slip a large, dry inverted test tube over it. Boil gently one minute. Keeping the large tube inverted, plunge its mouth under water in a dish. From the result deduce conclusions regarding (1) the solubility of ammonia in water, (2) the effect of heat upon its solubility.

EXERCISE 40

NEUTRALIZATION OF AMMONIUM HYDROXIDE BY ACIDS

(Ammonium Salts)

Apparatus. — 100 c.c. graduated cylinder. Hard glass tubing 1 cm. or more in diameter. Pipestem triangle. Porcelain crucible lid. Beaker. Evaporating dish. Glass rod (thin). Triangular file. Bottle.

Materials. — Red and blue litmus paper. Ammonium chloride. Ammonium sulphate. Ammonium nitrate. Concentrated hydrochloric acid. Ammonia-water.

Method. — See textbook, pages 236, 237, 238, 287, 302.

(a) Place in the graduated cylinder 10 c.c. concentrated hydrochloric acid. Read from the bottom of the meniscus (Fig. 4). Dilute to 100 c.c. with water and stir well. Transfer 20 c.c. of this dilute acid to a dry beaker and put the rest in a bottle in case of need.

Wash the cylinder and rinse it with a few drops of strong ammonia. Dilute 10 c.c. of ammonia to 100 c.c.

Add this dilute ammonia gradually to the 10 c.c. of dilute acid. Stir gently with a thin glass rod. Remember that the beaker is thin and can be easily perforated. Continually test whether you have added enough ammonia, by shaking most of the liquid off the rod and placing a very small drop on red

litmus paper. Use the same piece of paper for many tests. When the paper is turned blue (what does this indicate?), evaporate the liquid almost to dryness in a dish, using a very small flame toward the end. Let cool. What is the residue? Write the equation. How does the equation differ from the one which you wrote in (c) of the preceding exercise?

(b) Select a piece of hard glass tubing (Jena glass is best) 1 cm. or more in diameter, and cut a piece 20 cm. (8 in.) long. Cut it by making a deep notch by sawing with a triangular file, and bending away from the notch over the edge of the table. Place 1 c.c. of ammonium chloride in the middle of the tube. Introduce the ammonium chloride by means of a strip of paper 1 cm. (0.4 in.) wide which is folded along the center to make a V-shaped trough. Put in each end of the tube a piece of red and a piece of blue litmus paper. The litmus paper should be damp. Clamp the tube nearly but not quite in a horizontal position, putting the clamp 1 cm. from the end. Heat the ammonium chloride gently. Watch the litmus paper closely for changes, and for reversal of any changes which may occur. Does the litmus paper show that the salt is decomposed by heat into ammonia and hydrochloric acid? If so, does the sublimate in the tube prove that the two gases must, for the most part, reunite when they reach the cooler part of the tube?

Let the glass tube cool, wash it, and return it.

(c) Support a porcelain crucible lid on a pipestem triangle. Place on it a fragment of ammonium sulphate the size of the head of a pin, and heat. Result? Repeat with ammonium nitrate.

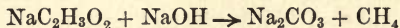
EXERCISE 41

METHANE

Apparatus. — Small test tube with delivery tube for collecting over water. Bottle. Mortar and pestle. Agate pan. Glass plate.

Materials. — Soda lime. Anhydrous sodium acetate (can be either purchased or made by heating the crystallized salt in an iron dish with constant stirring). Limewater.

Method. — Methane is obtained when a mixture containing sodium acetate and sodium hydroxide is heated:—



Fill the test tube two thirds full with a mixture of equal volumes of powdered soda lime and powdered anhydrous sodium acetate. Clamp in a horizontal position. Tap the tube with your pencil to make the powder settle and leave a channel along the top for the escape of gas. Heat gently. Avoid softening the glass by too high a temperature. Collect over water.

Record the physical properties of the gas. Burn a bottle of it and at once apply the limewater test. What product of combustion do you detect? What other substance must be formed by the combustion of methane?

EXERCISE 42¹

CARBOHYDRATES

Apparatus. — Wire gauze. Small test tubes. Beaker. Graduated cylinder. Thin glass rod.

Materials. — Glucose. Ordinary sugar. Molasses. Starch. Fehling's solution (see below). Sodium carbonate. Red litmus paper.

Method. — Glucose produces, with Fehling's solution, a red precipitate of *cuprous oxide*, Cu_2O .

(1) 69.28 grams of powdered copper sulphate ($\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$) is dissolved in warm water and 10 drops of sulphuric acid added. The solution is diluted to 1 liter in a graduated flask.

(2) 350 grams of Rochelle salt (potassium sodium tartrate, $\text{KNaC}_4\text{H}_4\text{O}_6$) and 100 grams of sodium hydroxide are dissolved together in water and the solution diluted to about 1 liter. This liquid must be kept in a bottle with a rubber stopper.

The mixture of equal volumes of (1) and (2) is called *Fehling's solution*. 1 c.c. of it interacts with 0.005 gram of grape sugar (glucose, $\text{C}_6\text{H}_{12}\text{O}_6$). The two solutions should be mixed just before use. Make 50 c.c. Fehling's solution by mixing 25 c.c. of each in a graduated cylinder, stirring thoroughly.

(a) In a test tube, dissolve about 0.5 c.c. of glucose in 20 c.c. water. Heat 10 c.c. Fehling's solution in a beaker to gentle boiling. Add two or three drops of the glucose solution. Result? The precipitate is cuprous oxide, Cu_2O . Add another drop of the glucose solution and boil again. Continue in this way until the *liquid over the precipitate* has become colorless. The *precipitate* will remain red. Be careful to add no more glucose solution than is necessary. What weight of glucose have you added?

(b) Dissolve about 0.5 c.c. cane sugar in 20 c.c. of water in a beaker. Add a drop or two of this solution to 5 c.c. Fehling's solution and heat in a test tube. Is there any result?

¹ The discussion of carbohydrates in textbook, Chapter 15, page 191, should be read before this exercise is carried out.

To the rest of the sugar solution, add 10 drops concentrated hydrochloric acid and heat on wire gauze for ten minutes to gentle boiling. As the water evaporates, add enough water to keep the volume at about 20 c.c. Cool and add solid sodium carbonate until a drop of the liquid makes a blue spot on red litmus paper. Then test with Fehling's solution in the same way as before. What does the result show about the action of acids on cane sugar? Would acetic acid have produced the effect as rapidly as hydrochloric? Why? The effect is due entirely to the H^+ ions.

(c) Boil 0.5 c.c. of starch powder with 20 c.c. of water and repeat (b) with the starch solution. What is the action of dilute acids on starch? What practical application is made of the fact? Why, in actual manufacture, is it sufficient to neutralize the hydrochloric acid with sodium carbonate, instead of removing it from the sirup (see textbook, page 193). Why does hydrochloric acid act on the starch more rapidly than most other acids? The effect is due entirely to the H^+ ions.

(d) Test a solution of 0.5 c.c. molasses in 20 c.c. water for glucose.

EXERCISE 43

ACETIC ACID, WOOD ALCOHOL, ACETYLENE

Apparatus. — Small test tubes. Bottle. Evaporating dish.

Materials. — Acetic acid. Sodium acetate. Iron filings. Zinc dust. Magnesium ribbon. Alcohol. Concentrated sulphuric acid. Red and blue litmus paper. Wood alcohol. Asbestos fiber. Copper wire No. 20. Iron wire. Limewater. Calcium carbide.

Method. — See textbook, pages 183, 201, 202, 353.

(a) Take 10 c.c. of acetic acid in a small test tube. Record its physical properties. Note the odor. Does it resemble that of any common substance? Add a few drops to a test tube full of water. Taste the dilute acid and test it with red and blue litmus paper. Place in one small test tube 1 c.c. of zinc dust, in another, the same volume of iron filings, and in a third,

a spiral made of 4 cm. of magnesium ribbon. Cover the metals with concentrated acetic acid. If necessary, heat very gently—not to boiling—with a small flame. What gas would you expect to obtain? Is the change rapid and violent or slow and moderate?

Acetic acid is an *inactive* acid. Compare its activity with that of an *active* acid (sulphuric), thus: Dilute 1 c.c. of acetic acid with 4 c.c. of water. In another tube, place 4 c.c. of water and add 1 c.c. of sulphuric acid. Add 1 c.c. of zinc dust to each and compare the behavior. The same kind of chemical process occurs in both tubes. The main difference is in the speed.

(b) Place 1 c.c. of sodium acetate in a test tube, add 2 c.c. of water and 1 c.c. of concentrated sulphuric acid. Warm gently. Odor? Effect on blue litmus paper? In this experiment, sulphuric acid produces acetic acid by interacting with sodium acetate.

(c) To 1 c.c. of sodium acetate in a test tube add 2 c.c. alcohol and 1 c.c. concentrated sulphuric acid. Warm gently for an instant—*not to boiling*. (*Care!*) Odor? The fragrant substance is called *acetic ester*. It has some important applications; for instance, in the manufacture of smokeless powder from guncotton. Its formation, in the way you have just carried out, serves as a *test* for acetic acid or an acetate.

(d) **Wood alcohol and formaldehyde.**—Note the odor of wood alcohol and compare it with that of grain alcohol. The odor is due to *acetone*, an impurity which is always present in commercial wood alcohol.

Dip a mass of asbestos 1 cm. (0.4 in.) in diameter into wood alcohol, place it on the base of your stand and set fire to it. Hold over the flame a dry, cold, clean bottle. One product of the combustion condenses on the walls. What is it? To identify the other product, pour at once into the bottle 5 c.c. of limewater, cover with the band and shake. Result?

Formaldehyde results from the imperfect combustion of wood alcohol. Make a spiral by wrapping 30 cm. of No. 20

copper wire around a lead pencil. Heat the spiral red hot, holding it with iron wire and hold it close to the surface of 2 c.c. of wood alcohol in a small test tube. The sharp odor is due to formaldehyde.

(e) **Acetylene.**

Fill a small test tube completely with water, cover tightly with the thumb, and invert it in an evaporating dish full of water. Slip under the mouth of the tube a 5 mm. (0.2 in.) bit of calcium carbide. If necessary, use another bit to complete the filling of the tube.

The solid product in the dish is *slaked lime* (calcium hydroxide). Test it with litmus paper (both kinds).

Remove the test tube from the dish, using the thumb, and burn the gas. At once pour into the tube 5 c.c. of limewater, cover with the thumb, and shake. What product of combustion do you detect?

EXERCISE 44

CHLORINE¹

Apparatus. — A 300 c.c. flask with doubly perforated rubber stopper, carrying a dropping funnel and a delivery tube bent twice at right angles (Fig. 20). Kipp hydrogen generator (one will serve for the class). Four bottles. Small test tube with cork. Wire gauze. Glass rod. Four glass plates. Iron spoon.

Materials. — Potassium permanganate (commercial). Charcoal. Red and blue litmus paper. Colored calico or other cotton fabric. Fresh slaked lime. Ink. Christmas-tree candles. Iron wire No. 20.

Method. — See textbook, pages 212, 213, 220.

Carry out this experiment under the hood. Avoid inhaling the gas. Be sure that the cork of the apparatus fits tightly. If the throat becomes

¹ This exercise is given because it is included in the syllabi of College Entrance Requirements. It may be dangerous in unskilled hands, and it is certain to cause great discomfort to the students. Many teachers will prefer to show it on the lecture table. However, the production of the gas is under perfect control and the method is greatly superior to the use of manganese dioxide and hydrochloric acid.

irritated, place some alcohol in a beaker or on a handkerchief and inhale the vapor.

If hoods are not available, this work should be omitted or done at a time when the windows of the laboratory can be open. A little ammonia sprinkled on the floor will reduce annoyance from chlorine.

Place crystals of potassium permanganate 1 cm. deep in the flask. The substance need not be pure. Fit the flask with a rubber stopper carrying a dropping funnel and a delivery tube. Support it firmly on wire gauze, and put a small flame 8 cm. below it. Allow hydrochloric acid to fall drop

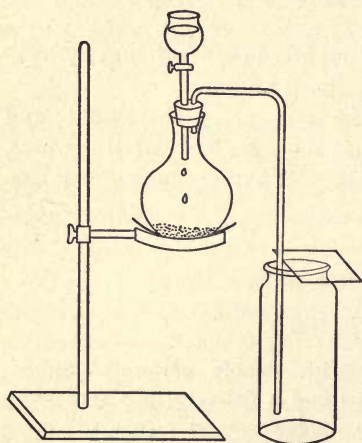


FIG. 20.

by drop into the flask. Collect by downward displacement one small test tube and four bottles full of the gas. Judge when the vessel is full by the color, and *immediately* cover it tightly with a glass plate and substitute another; otherwise the excess of chlorine will be forced out at the top and make the air unfit to breathe. Test tubes full of chlorine can be corked. The exit tube of the

chlorine apparatus must reach to the *bottom* of the vessel. This tube should be cut about a foot from the desk and united again by a short piece of rubber tube, that it may be moved without disturbing the apparatus. The vessel in which you are collecting chlorine should be kept covered, and the exit tube slipped between the cover and the side of the bottle. The bottles of chlorine must be kept covered during all experiments. When you have finished and desire to get rid of the chlorine, place the bottles under the hood, remove the covers without breathing, and at once retire to a distance. After the chlorine has escaped — which will require ten minutes — the bottles can be cleaned.

Record the physical properties of the element. Invert a test tube full in water and shake gently. Is it soluble?

Lower¹ a hydrogen flame into a bottle of chlorine. If you use a gas-generating bottle in this experiment, remember that it must be allowed to run five minutes and that the generator and stopper must be wrapped in a towel before lighting the hydrogen. Describe the appearance of the flame of hydrogen burning in chlorine. Hold a glass rod wet with ammonia near an open bottle of hydrochloric acid. Result? This is a *test* for hydrochloric acid. Now hold a rod wet with ammonia in the gas left in the jar in which the hydrogen was burned. Is it hydrochloric acid? Has it the color of chlorine? Heat a piece of charcoal in a spoon until it begins to burn, and place it in chlorine. Result? Keep the bottle covered.

Take out the charcoal and lower a lighted candle supported on a wire into the same bottle of chlorine. The candle is composed chiefly of compounds of hydrogen and carbon. The black substance thrown off from the flame is carbon (soot). Use the two preceding results to explain this one. What else must be produced? Let the covered jar stand till the soot settles and try the test with the glass rod wet with ammonia.

In the third bottle, place a bit of red litmus paper, a piece of blue litmus paper, a strip of colored calico, and a fragment of printed matter smeared over with writing ink until it is illegible. Describe and explain the results. What is the most important use of chlorine?

In the fourth bottle, place some fresh slaked lime (milk of lime answers well), and immediately cover the bottle tightly with the palm of the hand and shake it. Result? Does the chlorine disappear?

Carefully add nitric acid, a few drops at a time, to the contents of the bottle. What happens? What very important technical process does this illustrate? See textbook, page 317.

¹ Many teachers will prefer to omit this experiment or to show it on the lecture table.

EXERCISE 45

THE ACTION OF SULPHURIC ACID ON CHLORIDES

Apparatus. — Four small test tubes. Glass rod.

Materials. — Sodium chloride. Potassium chloride. Ammonium chloride. Barium chloride. Red and blue litmus paper.

Method. — See textbook, page 222.

Place 1 c.c. of sodium chloride in a small test tube. Do the same with the other chlorides, using a different tube for each. Drop into each tube 1 c.c. of concentrated sulphuric acid.

To show the behavior of water vapor with the gas given off, blow across the mouth of the tube. Ascertain its reaction with ammonia by holding a glass rod wet with this substance in the tube. Remember, however, that *any acid vapor or gas will give a white cloud with ammonia*. In writing the equation remember, also, that it is the gas (NH_3) given off by the ammonia, and not the dissolved substance, which combines with the hydrochloric acid.

Hold in each tube 1 sq. cm. of red and of blue litmus paper. Result?

EXERCISE 46

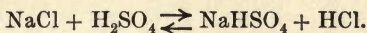
THE ACTION OF SULPHURIC ACID ON SALT

Apparatus. — 300 c.c. flask, with singly perforated rubber stopper and delivery tube bent twice at right angles. Beaker. Two small, dry test tubes. Three dry bottles. Agate pan. Glass rod. Wire gauze.

Materials. — Table salt. Red and blue litmus paper. Christmas-tree candles. Iron wire. Filter paper.

Method. — See textbook, page 223.

Note the *equation* : —



Place about 25 c.c. of water in a beaker. Stand the beaker in a pan of cold water and slowly pour in 50 c.c. concentrated sulphuric acid, stirring constantly. Cool *completely* and pour the liquid upon 30 grams of salt in the flask.

Place the flask on wire gauze and apply a gentle heat. Collect, by downward displacement, two test tubes and three bottles of hydrogen chloride. The delivery tube must reach to the bottom of the bottle, and the bottle and delivery tube must be *dry*. Why?

Is hydrogen chloride soluble in water? Try it by placing a test tube full with its mouth downward in water. Does the gas burn? Try with a test tube full. Does it support the combustion of a candle? How does it affect red and blue litmus paper?

Pour some ammonia on a wad of filter paper and throw it into a bottle of hydrogen chloride. Result?

EXERCISE 47

THE ACTION OF HYDROCHLORIC ACID ON SODIUM
HYDROGEN SULPHATE

Apparatus. — Small test tube. Lens.

Material. — Sodium hydrogen sulphate, NaHSO_4 .

Method. — See textbook, page 222.

Make 5 c.c. of a concentrated solution of sodium hydrogen sulphate and add concentrated hydrochloric acid gradually.

Examine with a lens. The interaction of salt with sulphuric acid, or of hydrochloric acid with sodium hydrogen sulphate, is *partial*. It stops when all four substances, in certain fixed quantities, are present in each c.c. of the liquid. In Exercise 45, the escape of the hydrochloric acid removes it from the sphere of the change and causes the reaction to proceed to completion in one direction. In this exercise the insolubility of salt in hydrochloric acid takes the salt out of the conflict and pushes the reaction to completion in the other direction.

EXERCISE 48

THE ACTION OF HYDROCHLORIC ACID UPON
METALS

Apparatus. — Beaker. Eight small test tubes. Medicine dropper. Watch glass.

Materials. — (a) Sodium. (b) Zinc (granulated and dust). Sheet copper. Aluminium turnings. Test lead. Iron filings. Magnesium ribbon. Mercury.

Method. — See textbook, page 219, section 301.

(a) Drop a 2 mm. (no larger) piece of sodium into 5 c.c. of concentrated hydrochloric acid in a small beaker. Does hydrogen escape? Hold a burning match close to the globule. Why is the flame of the burning gas yellow?

(b) Dilute 20 c.c. of concentrated hydrochloric acid with 20 c.c. of water. Place in separate test tubes 1 c.c. of granu-

lated zinc, zinc dust, aluminium turnings, iron filings, and test lead. In a sixth tube place 1 sq. cm. of sheet copper, in a seventh, 3 cm. of magnesium ribbon coiled into a spiral, and in an eighth, 1 c.c. of mercury, using a medicine dropper. Add to each tube 5 c.c. of the mixture of hydrochloric acid and water.

Notice and record any change in each tube. If no change is evident, heat gently, but remember that gas escaping *while the flame is used* may be steam or hydrogen chloride. If the escape of gas continues while the flame is withdrawn, there is evidence that the acid and the metal are interacting. Hydrogen can be identified by burning it, but remember that, *when much diluted with other gases*, it does not burn. Does the hydrogen come from the metal or the acid? Arrange the metals in a list, beginning with the most active.

When interaction occurs, there must be another product. What must it be in each case?

In order definitely to prove the formation of the second product, evaporate one of the solutions to dryness in a watch glass laid upon a beaker half full of water, which is gently boiled. Wait until the action is over, and filter, if necessary, before evaporating.

Did you notice any difference in the behavior of zinc dust and granulated zinc? If so, what is the cause?

EXERCISE 49

THE ACTION OF HYDROCHLORIC ACID UPON
OXIDES

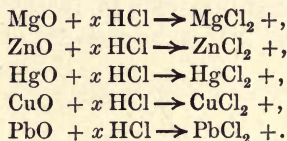
Apparatus. — Two watch glasses 8.5 cm. in diameter. Five small test tubes. Beaker. Funnel.

Materials. — Magnesium oxide. Zinc oxide. Mercuric oxide. Cupric oxide. Lead oxide. Filters.

Method. — See textbook, page 221.

Place 1 c.c. of magnesium oxide in a small test tube. Do the same with the other oxides, using a separate tube for each. Add to each tube 2 c.c. of water and 2 c.c. of conc. hydrochloric acid. Warm gently, but not to boiling. Is gas evolved? Be careful to distinguish between mere boiling and the escape of gas. The former ceases when the burner is removed—the latter continues. What becomes of the hydrogen of the acid and the oxygen of the oxide? What, then, must be the two products in each case?

Complete the following equations, supplying coefficients where needed:—



Prove by evaporating some of the filtered liquids, as in the preceding exercise, that these chemical changes occur. Use a new filter for each substance and wash the funnel each time before using it again. If time presses, the five evaporations can be distributed to different students.

Contrast the behavior of hydrochloric acid with mercury and with mercuric oxide (Exercise 48).

Contrast the behavior of hydrochloric acid with lead and with lead oxide.

Contrast the behavior of hydrochloric acid with copper and with copper oxide.

Contrast the behavior of hydrochloric acid with zinc and with zinc oxide. Explain.

EXERCISE 50

FLAME TESTS

Apparatus. — Wire cutter. Cobalt glass. Two small test tubes.

Materials. — Nitrates of calcium, barium, strontium, sodium, and potassium. The chlorides can be used instead. Solution of lithium chloride. Iron wire No. 20.

Method. — See textbook, pages 209, 241, 262, 312.

Obtain, on five small pieces of paper, 0.5 c.c. (*not more*) of each of the nitrates mentioned. Take in a clean test tube 1 c.c. (*not more*) of lithium chloride solution. Make a 2 mm. loop on the end of a 15 cm. (6 in.) piece of iron wire. Dip the loop and the adjacent part of the wire into a test tube (*not the bottle*) containing 5 c.c. of concentrated hydrochloric acid, and hold it in the blue flame of the burner until it gives no color to the flame. Clean the wire in this way after each test, before using it again. If you find it impossible to get the wire so clean that it gives no color to the flame, discard it and get a fresh piece. Discard the acid at the end of the work.

Take up a trace of calcium nitrate on the loop, hold it in the lower, outer part of the flame, observe and record the color. Do the same with each of the others. The wire may be moistened when dipped into the salt. Or, if the wire is red hot, enough of the salt will usually adhere to it to give the result.

Look at the potassium flame and also at the sodium flame through cobalt glass. Which kind of light is quenched by the glass and which transmitted? Does this suggest a method of detecting potassium in the presence of sodium? Mix the nitrates of the two metals and look at the flame color of the mixture without the glass. Which metal do you detect? Now use the glass. Result? If a spectroscope is at hand, use it to examine the spectrum of sodium.

EXERCISE 51¹

PREPARATION OF SODIUM HYDROGEN CARBONATE AND SODIUM CARBONATE

(Solvay Process)

Apparatus. — Gas-generating bottle with two-hole rubber stopper bearing thistle tube and delivery tube, bent twice at right angles. Test tube with one-hole rubber stopper and delivery tube. Iron dish (porcelain will answer, with care). Graduated cylinder. Trip scales. Large test tube with cork. Large test tube.

Materials. — Table salt. Ammonium carbonate. Marble chips. Sodium hydrogen carbonate. Filters. Manila paper. Limewater.

Method. — See textbook, page 323.

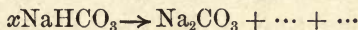
(a) Place 25 c.c. strong ammonia and 15 c.c. water in the large test tube and add 8 grams of ammonium carbonate (powdered). Cork and shake until the latter is dissolved. Add an excess of sodium chloride and shake persistently until the liquid is saturated.

Decant the clear liquid into a test tube and pass in carbon dioxide in a slow stream for half an hour, or until an abundant precipitate of sodium hydrogen carbonate separates. Meanwhile go on with (b) or (c). The carbon dioxide is made from marble and hydrochloric acid (Exercise 21). Cover the marble with water and add concentrated hydrochloric acid until you obtain a steady evolution of gas. When the current of gas slackens, add fresh acid. Filter the sodium hydrogen carbonate and dry it between folds of manila paper. Note its appearance and taste.

(b) Transformation of sodium hydrogen carbonate into sodium carbonate.

¹ In order to finish in time, it is necessary to assign (a), (b), and (c) to different groups of students. Or (a) may be carried out one week and (b) and (c) the next, the sodium hydrogen carbonate being dried in the meanwhile. The time required to saturate with carbon dioxide can be reduced by drawing out the end of the delivery tube so as to produce small bubbles.

Place 2 c.c. sodium hydrogen carbonate in a test tube and support in a horizontal position. The delivery tube dips into limewater. Heat gently, not hot enough to color the flame yellow. What gas escapes? What condenses in the cool part of the tube? Complete the equation:—



Compare the taste of the product with that of sodium hydrogen carbonate.

(c) Weigh 10 grams of sodium hydrogen carbonate in a weighed iron or porcelain dish. Heat at first gently, then intensely. Let cool and reweigh. Compare the taste of the product with that of sodium hydrogen carbonate. What is the loss in weight? Complete the equation in (b). Calculate from the completed equation the amount which 10 grams should lose. What is the error of your determination?

EXERCISE 52

HYDROGEN FLUORIDE

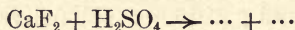
Apparatus.—Lead dish. Forceps. Glass rod. Agate pan.

Materials.—Powdered fluorspar (calcium fluoride, CaF_2). Red and blue litmus paper. Paraffin. Glass plate. Pins.

Method.—See textbook, page 263.

Place 2 c.c. of powdered fluorspar in the lead dish, moisten but do not cover it with concentrated sulphuric acid, support the dish 5 cm. (2 in.) above a small flame, and allow it to get warm. Wet a glass rod with ammonia and hold it over the dish. Do not let any ammonia fall into the dish. Remembering the behavior of ammonia with hydrogen *chloride* (see textbook, page 236), explain its behavior with the hydrogen *fluoride*, HF. Write the equation for the combination of ammonia and hydrogen fluoride and name the product. Hold over the dish a piece of red and a piece of blue litmus paper. Both should be damp.

Complete the equation : —



What is the advantage of using an acid like sulphuric, which does not vaporize easily, in this experiment? Why would it be impossible to use hydrochloric or nitric acid, although both are more active than sulphuric? Compare textbook, page 297.

Support a glass plate *10 cm. (4 in.) above a small flame*. Rub the upper side of the warm plate with a piece of paraffin until you have covered it evenly. Let cool. Write the name of the element which is combined with hydrogen in hydrogen fluoride upon the plate with a pin, being careful to expose the glass. Write also the year of the isolation of fluorine and the name of the great French chemist who first isolated it. (See textbook, page 264.) Cover the dish with the plate, paraffin side down, in such a way that your writing is completely exposed to the gas in the dish. Let stand for half an hour or more. The warmer the place, the better. Do not heat with the flame. Drop the plate into a pan of boiling water to remove the paraffin. Take it out with forceps, wipe, and examine.

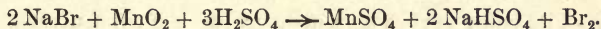
EXERCISE 53

BROMINE

Apparatus. — Large test tube. Four small test tubes. Glass rod.

Materials. — Potassium bromide (powder). Manganese dioxide (powder). Potassium iodide solution. Chloroform. Ether. Carbon disulphide. Bromine. Red and blue litmus paper. Potassium iodide starch paper.¹

Method. — See textbook, pages 260, 261, 262. Note the *equation* : —



(a) Place 1 c.c. water in a small test tube. Add 2 c.c. concentrated sulphuric acid. Cool. Mix 1 c.c. potassium bromide powder with 2 c.c. manganese dioxide powder on paper and place in another test tube. Add enough of the diluted sulphuric acid to moisten the mass. Warm gently. Bromine vapor escapes. Note color and odor. (*Care!*) Hold in the vapor a drop of potassium iodide solution (glass rod). If the color in the drop is due to liberated iodine, what else must be formed at the same time?

Hold a piece of potassium iodide-starch paper in the vapor. Potassium iodide does not color starch, as the whiteness of the unused paper proves. Iodine, on the other hand, colors starch deep blue (delicate test both for iodine and starch). Explain, then, the behavior of the paper in bromine vapor.

Remove the flame and stop the production of bromine by filling the tube with water. Other soluble bromides would behave like potassium bromide in this experiment, and other soluble iodides like potassium iodide. The potassium salts are used merely because they are most easily obtained.

CAUTION: Ether and carbon disulphide must not be used in the vicinity of flame. Bottles containing them must be kept stoppered.

¹ To make this, boil 1 c.c. starch powder with 100 c.c. water, add a 1 mm. crystal of potassium iodide, dip strips of filter paper 1 cm. wide in the liquid, and hang them over a piece of glass tubing to dry.

(b) Add one drop of bromine to 15 c.c. of water in a small test tube. Shake the liquid in the test tube. The solution you have made is called *bromine water*. Place 5 c.c. of it in each of the three small test tubes.

CAUTION: Do not withdraw the stopper of the bromine bottle altogether. Loosen it and let the liquid drip out between stopper and bottle. Use the opportunity to notice the high density of bromine. Avoid inhaling bromine vapor.

To one tube, add 2 c.c. of ether, cover with the thumb and shake. The color serves as a measure of the concentration of the bromine in the two layers. What happens to the bromine? Note that the bromine does not pass *completely* from the water to the ether layer. Whether there is much or little bromine present, it is divided between the water and the ether so that the value of the fraction —

$$\frac{\text{bromine in 1 c.c. water layer}}{\text{bromine in 1 c.c. ether layer}}$$

is always the same and very much less than unity.

If you had to extract the bromine as completely as possible from a liter of bromine water, and could use only 500 c.c. of ether, how would you go about it? Would it be best to use the ether all at once? Or could you get a better result by shaking up with a small portion of ether first, separating the ether solution of bromine, shaking up the water again with another small portion of ether, and so on? Why? (Compare textbook, page 31, section 40.)

Try, in the second tube, 2 c.c. of chloroform, and in the third, 2 c.c. of carbon disulphide. Cover and shake as before. In these cases, also, the water competes with the other liquid for the bromine.

(c) **Hydrogen bromide.** — Place 1 c.c. of potassium bromide crystals in a test tube and cover with a mixture of 3 volumes of sulphuric acid and 1 volume of water. Use a gentle heat. Try the action of the gas upon red and blue litmus paper.

Hold a drop of ammonia in the gas. Make a comparison between hydrogen bromide and hydrogen chloride.

Pure hydrogen bromide is colorless. Had the gas any color in this experiment? Recalling the color of bromine vapor, what did the color of your gas in this experiment indicate? Draw a conclusion regarding the stability of hydrogen bromide as compared with hydrogen chloride. Hydrogen iodide (HI) cannot be prepared at all by the action of sulphuric acid on iodides, because it is, for the most part, decomposed with liberation of iodine. (See textbook, page 259.) How about its stability? Inquire of the instructor about the stability of the hydrogen compounds of this series and read up the matter in the textbook, page 267.

EXERCISE 54

IODINE

Apparatus. — Watch glass (large enough to cover evaporating dish). Graduated 100 c.c. cylinder. Evaporating dish. Two large test tubes, one of which must be dry. Four small test tubes.

Materials. — Potassium iodide. Iodine. Manganese dioxide (powder). Starch solution. Alcohol. Ether. Carbon disulphide. Chloroform. Filter paper.

Method. — See textbook, pages 258, 259, 260. Note the *equation* : —



CAUTION: *Do not allow iodine to come into contact with the skin.*

(a) Place 1 c.c. of water in a small test tube and add 2 c.c. concentrated sulphuric acid. Cool. Mix 1 c.c. powdered potassium iodide with 2 c.c. manganese dioxide powder on paper. Transfer to a dry dish. Moisten the mixture with three to four drops of the diluted sulphuric acid. Cover the dish with a watch glass which is two thirds filled with cold water to keep it cool. Support the dish 10 cm. (4 in.) above a 2 cm. (0.8 in.) flame. When a sufficient sublimate collects on

the watch glass, extinguish the flame. Dip a strip of filter paper into starch solution and hold it in the vapor from the dish. Result? Examine the sublimate on the watch glass.

Iodine stains upon the hands can be removed by rubbing with a wet crystal of sodium thiosulphate (commonly called hyposulphite or hypo).

(b) Gently warm a large test tube and throw in a crystal of iodine. Notice color and odor of iodine vapor. Invert the tube and notice the high specific gravity of iodine vapor. If the formula of iodine is I_2 , what would the specific gravity of the vapor be, referred to oxygen? Referred to air?

Use the same test tube to study the solubility of iodine in alcohol. Take one crystal of iodine and 3 c.c. alcohol. Shake. This alcoholic solution of iodine is called *tincture of iodine* in pharmacy.

CAUTION: Ether and carbon disulphide must not be used near a flame.

Fill a large test tube one half with water, add three crystals of iodine, warm, —not quite to boiling-point— and shake. Judge of the solubility of iodine in water by the color of the liquid. Place 10 c.c. of the clear solution in each of four small test tubes. Use three of these to ascertain the effect of shaking (1) with 2 c.c. ether, (2) with 2 c.c. chloroform, (3) with 2 c.c. carbon disulphide. Discuss the results from the same point of view as the similar experiments with bromine (Exercise 53).

To the fourth tube, add 10 c.c. starch solution. Mix thoroughly. Reserving the remainder of the blue liquid, pour 1 c.c. of it into a clean graduated cylinder. The cylinder now contains 0.0001 gram of iodine. Add water to the 100 c.c. mark and mix thoroughly by covering the cylinder with the hand and upsetting it several times.

Now pour off half the liquid, fill again to 100 c.c., and mix. Place the cylinder on a piece of white paper and look down through it. How much iodine does the cylinder now contain? Proceed in this way until it becomes impossible to perceive the color.

What is the smallest quantity of iodine which you can detect by means of the test with starch. Is the test delicate?

Heat the remainder of the original mixture of iodine and starch solution nearly to the boiling point (80°). Result? Cool at once in a stream of water. Result?

EXERCISE 55

SILVER COMPOUNDS OF THE HALOGENS

(The Replacement of One Halogen by Another)

Apparatus. — Porcelain dish. Six small test tubes.

Materials. — Chlorine water. Bromine water. Potassium bromide. Potassium iodide. Carbon disulphide. Potassium chloride. Silver nitrate solution. Salt. Mossy zinc. Distilled water.

Method. — See textbook, pages 260 and 261.

CAUTION: *Do not allow silver nitrate or its solution to come into contact with the skin.*

(a)¹ Dissolve 0.5 c.c. sodium chloride in 5 c.c. distilled water and add 3 c.c. silver nitrate solution. Shake vigorously. Transfer the precipitate, by rinsing with water, to a dish, pour off the water, add a 0.5 cm. bit of mossy zinc and one drop of concentrated sulphuric acid. Cover with a paper to exclude the light and examine from time to time. The visible product is *silver*. What else must be formed? Notice that the change spreads through the mass and that the silver chloride is all converted into silver, although most of it never touches the zinc at all. This might be called chemical action at a distance. It can be readily explained, using the ideas of the ionization hypothesis, but the full explanation lies beyond the scope of this book. Remove the unused zinc, wash the silver well with water, and ask the instructor what to do with it.

(b) Dissolve a 1 mm. crystal of potassium chloride in 5 c.c.

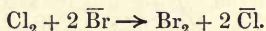
¹ (a) and (b) should be started at the beginning of a laboratory period and allowed to stand until near the end.

of distilled water in a small test tube. In two other tubes, dissolve 1 mm. crystals of potassium bromide and potassium iodide each in 5 c.c. distilled water. Add to each tube 1 c.c. silver nitrate solution and shake. Names and formulas of the precipitates? Equations? Use half of each precipitate to study the solubility in ammonia. Result? Stand the other half in bright sunlight or the brightest light available as long as possible. Result?

CAUTION: Carbon disulphide must not be used near a flame.

(c) Dissolve a 1 mm. crystal of potassium bromide in 5 c.c. water in a small test tube. Add 2 c.c. carbon disulphide and shake. Does chemically combined bromine behave like free bromine toward carbon disulphide? To discuss the result accurately, we should use the language of the hypothesis of ionization. The water solution of potassium bromide contains *bromine ions* ($\overline{\text{Br}}$). These are colorless. Also they are held tenaciously by the water and scarcely enter the carbon disulphide at all.

Now add 3 c.c. chlorine water and shake. Result? The result can be summed up in the statement that the chlorine combines with the potassium, setting free the bromine, which dissolves in the carbon disulphide. Speaking in terms of the ionization-idea, the chlorine (Cl_2) takes the negative charges from two bromine ions, producing two chlorine ions ($\overline{\text{Cl}}$) and bromine (Br_2), which dissolves in the carbon disulphide.



The result may be said to prove that the "affinity" of chlorine for potassium is greater than that of bromine. It would be more exact to say that it shows that the tendency of chlorine to exist as an ion is greater than that of chlorine.

(d) Repeat (c), using a 1 mm. crystal of potassium iodide, instead of the potassium bromide. Which has the "strongest affinity" for potassium, chlorine or iodine? More exactly, which has the strongest tendency to exist as ion?

(e) Repeat (c), using a 1 mm. crystal of potassium iodide

with the water and carbon disulphide as before, but, instead of the 3 c.c. chlorine water, add 0.5 c.c. bromine water and shake. Which has the "strongest affinity" for potassium, bromine or iodine? Which has the strongest tendency to exist as ion?

EXERCISE 56

NEUTRALIZATION (1)

Apparatus.—Beaker. Porcelain dish. Lens. Glass rod.

Materials.—Sodium hydroxide. Potassium hydroxide. Red and blue litmus paper.

Method.—See textbook, pages 245 and 246.

CAUTION: *Do not allow potassium hydroxide or sodium hydroxide to come into contact with the skin.*

(a) Let concentrated hydrochloric acid fall, drop by drop (*Care!*), upon a 2 cm. (0.8 in.) piece of sodium hydroxide in a beaker. It is best to withdraw the stopper partially and allow the acid to leak out between the stopper and the bottle. From time to time put a small drop of the liquid upon red and a drop upon blue litmus paper, using a thin glass rod. Use the same paper for many tests.

When the stick is disintegrated and the change seems *complete* (acid reaction to litmus), scrape the solid product into a dish and dry it with a small flame kept in motion. When entirely dry and odorless, let cool and taste. Note shape of the crystals with a lens or microscope. What is the solid? What must have been the second product? Write the equation.

(b) Let concentrated nitric acid fall, drop by drop, upon a 2 cm. piece of potassium hydroxide (*Care!*) until the change is complete (litmus paper). Dissolve the result in the smallest possible quantity of hot water and let cool. Shape and appearance of the crystals? Equation?

EXERCISE 57

NEUTRALIZATION (2)

(Quantitative)

Apparatus. — Burette. Graduated cylinder. Balance or trip scales. Beaker. Porcelain dish. Thin glass rod.

Materials. — Normal acid.¹ Sodium hydroxide. Potassium hydroxide. Litmus solution. Cochineal solution.

Optional. — Washing soda. Commercial potassium carbonate. Limewater.

Method. — See textbook, page 256.

Read up the subject of neutralization and normal acids in the textbook, page 256. One cubic centimeter of a normal acid neutralizes the molecular weight in milligrams of sodium hydroxide or potassium hydroxide, but only *one half* the molecular weight of sodium carbonate or potassium carbonate.

Clamp the burette in a vertical position. Note that it reads downward. Thus, when halfway between 9 and 10, the reading is 9.5 c.c., not 10.5. If the burette is wet, rinse it out with a few drops of the normal acid and discard the latter. *Dry* beakers must be used in handling the normal acid, to avoid diluting it.

(a) Dry a dish, weigh it, and weigh in it 4 grams of sodium hydroxide. Add 50 c.c. water and dissolve the sodium hydroxide by stirring, using a gentle heat if necessary. Transfer to a graduated cylinder and rinse the dish into the cylinder three times with small quantities of water. Dilute to 100 c.c. *Mix thoroughly.*

¹ Most quickly made by dissolving 63 grams oxalic acid ($C_2H_2O_4$, 2 H_2O) to a liter. Trip scales are accurate enough for the weighing. It is well to have at least 75 c.c. for each student; more, if the work is extended to cover sodium carbonate, pearlash, etc. The student can readily make it himself in the graduated cylinder, but the exercise then becomes too long for the usual laboratory period. Use cochineal as indicator in presence of carbonic acid. Digest a 1 cm. layer of the crushed insects under half a liter of alcohol with occasional shaking. Other indicators may be substituted. Mohr burettes are best suited to elementary work.

Pour exactly 20 c.c. of the solution of sodium hydroxide into a clean beaker, and add enough litmus solution to give a distinct but not a strong color (about five drops). Fill the burette to zero with the normal acid, making sure that the portion below the pinchcock is also filled. Stand the beaker on a piece of white paper (filter paper) and run in the normal acid — 10 c.c. can be added rapidly; then add one drop at a time, stirring constantly, until the color change occurs. Calculate the percentage of sodium hydroxide in the sample. Remember that 20 c.c. contains $\frac{1}{5}$ of the quantity weighed out. Thus, suppose that 19 c.c. of the normal acid were required. Since each cubic centimeter of the normal acid consumes 40 milligrams of sodium hydroxide, the quantity of the latter present must have been, in grams, $0.040 \times 19 = 0.76$ gram NaOH. The 20 c.c. of sodium hydroxide solution used contained $4 \times \frac{1}{5} = 0.8$ gram commercial NaOH. So the percentage of true sodium hydroxide must be: —

$$\frac{0.76 \times 100}{0.8} = 95 \text{ per cent, the balance being impurities.}$$

Repeat, using 25 c.c. of the sodium hydroxide, colored with 10 drops of cochineal solution instead of litmus. Does the volume of sodium hydroxide solution taken affect the result? Does the nature of the indicator affect it? Remember that 25 c.c. contains $\frac{1}{4}$ of the quantity of sodium hydroxide you weighed out.

If the sodium hydroxide had been perfectly pure, what special concentration would have been obtained by using 4 grams of it in 100 c.c.? To what extent did your solution depart from this concentration?

(b) Weigh in a dish 5.6 grams potassium hydroxide and repeat the whole experiment, making the same calculations and answering the same questions.

In (c) and (d) use cochineal and omit the experiment with litmus.

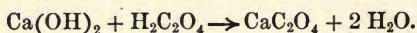
(c) Make the same experiment with 14.3 grams of washing

soda crystals. Calculate the percentage of sodium carbonate, Na_2CO_3 . How close is your result to that calculated from the formula $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$?

(d) Make the same experiment with 6.9 grams commercial potassium carbonate (pearlash). Calculate the percentage of potassium carbonate, K_2CO_3 .

(e) Measure 100 c.c. of limewater into a beaker, color with litmus or cochineal, and add normal acid from the burette until the change in color takes place. Be careful, as only a small volume of acid will be needed. Since the molecular weight of $\text{Ca}(\text{OH})_2$ is 74, 1 c.c. of normal acid equals $\frac{74}{2}$ or 37 milligrams of slaked lime. Calculate the weight of calcium hydroxide in 100 c.c. of limewater.

According to the most exact figures obtainable, 100 c.c. limewater at room temperature (20°C .) contains 0.165 gram calcium hydroxide. What was the error of your result? The white precipitate formed is calcium oxalate:—



EXERCISE 58

THE ACTION OF BASES ON SALTS

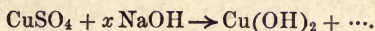
Apparatus.—Beaker. Small test tubes. Mortar and pestle.

Materials.—Sodium hydroxide. Copper sulphate. Silver nitrate. Mercuric chloride. Zinc sulphate. Lead nitrate. Aluminium sulphate. Ferrous sulphate. Mercuric oxide.

Method.—The hydroxides of the heavy metals (see textbook, page 368) are mostly insoluble in water. They precipitate when sodium hydroxide solution is added to a solution containing a salt of the corresponding metal. Some insoluble hydroxides dissolve in excess of sodium hydroxide solution.

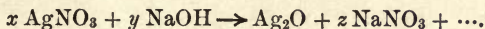
(a) Dissolve 10 grams of sodium hydroxide in 80 c.c. water in a beaker. Aid the solution by a small flame and gentle stirring. Cool the liquid. Dissolve 0.5 c.c. copper sulphate in 5 c.c. water in a small test tube. Cool, if you have used

heat. Add 5 c.c. of the sodium hydroxide solution. Complete the equation:—

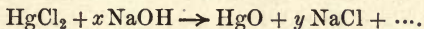


Heat the contents of the tube to gentle boiling. Result?

Repeat, using a 1 mm. crystal of silver nitrate instead of the copper sulphate. Boiling is necessary only in the case of copper sulphate. *Handle silver nitrate with paper*, not with the fingers. Complete the equation:—

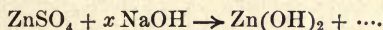


Repeat, using 0.5 c.c. of mercuric chloride. Complete the equation:—



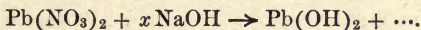
Compare this mercuric oxide with that in the stock bottle. Chemists are not yet agreed upon the cause of the striking difference.

(b) Dissolve 0.5 c.c. zinc sulphate in 5 c.c. water. Add sodium hydroxide solution, a few drops at a time, shaking well after each addition. When a considerable precipitate is obtained, stop to examine it and then continue the addition of sodium hydroxide until you have added 10 c.c. Complete the equation:—

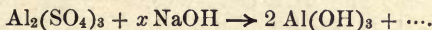


What happens when more sodium hydroxide is added to the precipitate?

Treat lead nitrate in the same way as zinc sulphate. Complete the equation:—



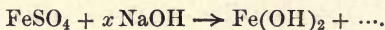
Treat aluminium sulphate in the same way as zinc sulphate. Complete the equation:—



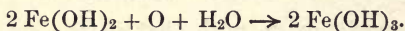
What important difference exists between the substances in (a) and those in (b)?

(c) Select a clear crystal of ferrous sulphate about 0.5 cm. in diameter and hold it in a stream of water until the surface is dissolved away. Dry with paper and crush in a clean mortar. Powder finely.

Boil 10 c.c. water in a small test tube to expel the dissolved air and cool in a stream of water. Add the ferrous sulphate and dissolve it by stirring. Do not heat. When completely dissolved, add 10 c.c. sodium hydroxide solution. The precipitate is ferrous hydroxide. Complete the equation:—



Shake the tube vigorously for five minutes and note any change in the precipitate. It passes into ferric hydroxide. The complete equation is:—



EXERCISE 59

THE ACTION OF A METAL ON A SOLUTION OF A SALT OF ANOTHER METAL

Apparatus.—Scissors. Tinner's shears. Lens. Ten small test tubes.

Materials.—(a) Sheet copper. Sheet zinc. Copper wire No. 30. (b) Silver nitrate. Mercuric chloride. Copper sulphate. Lead nitrate. Tin chloride (stannous). Zinc sulphate. Sandpaper. Dennison labels, small.

Method.—See textbook, page 293.

(a) Cut a piece of sheet copper 5×5 cm. (2×2 in.). Clean it with sandpaper and cut it into five strips each 5×1 cm. (2×0.4 in.). Prepare, in the same way, five strips of sheet zinc. 0.5 cm. from the end of each of the ten metal strips fasten one end of a copper wire 20 cm. (8 in.) long. The wire may be cut with ordinary scissors.

(b) 20 c.c. dilute solution of the salts listed in (b) is required. Prepare by dissolving 1 c.c. of the salt in 20 c.c. water.

Heat, if necessary, but cool before using. Use a *separate test tube for each salt*. Tin chloride will not dissolve clear in water alone. Add, therefore, to its solution a few drops of hydrochloric acid. Use only 0.5 c.c. of silver nitrate and of mercuric chloride. *Do not touch either of these two with the fingers*. Label each tube with the formula of the substance it contains and also with the symbol of the metal ion formed when the substance dissolves in water. Obtain this information from the textbook. Put on the label, also, the color of the solution.

Place 10 c.c. of each of the solutions, omitting the copper sulphate, in separate test tubes, and suspend in each a piece of sheet copper so that 1 cm. (0.4 in.) of the metal is above the surface.

In the same way, suspend a strip of zinc in another 10 c.c. of each solution, omitting the zinc sulphate. Let stand. After a time, withdraw the strips and examine them minutely with a lens. Use the upper portion, which should not have been wet with the liquid, for comparison. Be prepared for the fact that finely divided metals reflect light differently and present a very different appearance from the same metals in compact polished condition.

What appears to have happened? Is the deposit on the copper the only product? If not, what must be the second product, in each case? Note carefully any changes in the colors of the solutions. Do these changes confirm your conclusions? State your results in a table of four columns, putting in the first, the metal used, in the second, the substance in solution, in the third, the deposit, and in the fourth, the other product. Write an equation for each change, first in the ordinary way. Then write the more simple equations which describe the same changes in the light of the ionic theory.

EXERCISE 60

IONIZED AND UN-IONIZED SOLUTIONS

Apparatus. — Small test tubes, some of which must be dry.

Materials. — Cupric chloride. Cobalt chloride. Cupric sulphate. Cobalt sulphate. Alcohol (95 per cent). Solution of hydrogen chloride in toluene. Mossy zinc. Magnesium ribbon. Iron (small nail). Marble fragments.

Method. — See textbook, page 288.

Exercise unusual care to avoid waste of the cobalt salts. They are expensive. The solution of hydrogen chloride in toluene is easily made by passing the gas into toluene until the latter is saturated. It should be kept in a tightly stoppered bottle.

(a) Dissolve 0.2 c.c. of cupric sulphate in 2 c.c. water. Do the same with 0.2 c.c. cobalt sulphate and with 0.2 c.c. cobalt chloride in different tubes. Note the colors of the liquids. Other cupric and cobalt compounds in dilute water solution show *the same colors*, respectively. What conclusion can be drawn from this fact? The solutions of cupric compounds (which all have the same color) and the solutions of cobalt salts (which all have the same color) are *good conductors of the electric current*. What follows from this, regarding the condition of the salt? Does it confirm your conclusion drawn from the color?

Dissolve 0.3 c.c. cobalt chloride in 3 c.c. alcohol. Do the same, in a separate tube, with cupric chloride. Study the colors of the liquids and compare them with the water solutions containing the same metals. Does the color of cobalt chloride in alcohol resemble the color of water solutions of cobalt salts? Has the alcohol solution of cupric chloride the same color as cupric compounds dissolved in water? What conclusion do you deduce regarding the condition of the salts in the solutions?

These alcoholic solutions are all *nonconductors of the current*. Does this confirm your conclusion drawn from the colors? Can you think of any method, based upon a study of the boiling points of water and alcohol solutions, by which your conclusions might be tested?

Pour half of the alcoholic solution of cupric chloride into 3 c.c. water in a small test tube. Result? Do the same with the alcohol solution of cupric chloride in another tube. Result? *What is indicated by the fact that the color changes and the appearance of conductivity in the liquids are simultaneous?*

(b) Obtain, in a dry test tube, 5 c.c. of a solution of hydrogen chloride in toluene. Test the behavior of this liquid in dry test tubes with zinc, magnesium, iron (small nail), and a bit of marble or calcite. Result? Investigate the action of ordinary hydrochloric acid on the same substances. Result?

The water solution of hydrogen chloride conducts the electric current, but the toluene solution is a nonconductor. Explain the connection between chemical activity and conductivity.

EXERCISE 61

HYDROLYSIS

Apparatus. — Reading glass or other long focus lens. Five small test tubes. Glass rod.

Materials. — (a) Cupric chloride (CuCl_2). Stannous chloride (SnCl_2). Ferric chloride (FeCl_3). Cupric sulphate (CuSO_4). Aluminium sulphate ($\text{Al}_2(\text{SO}_4)_3$).

Distilled water. Red and blue litmus paper.

(b) Sodium carbonate (Na_2CO_3). Potassium carbonate (K_2CO_3). Sodium sulphide (Na_2S).

(c) Sodium chloride (NaCl). Potassium chloride (KCl). Sodium nitrate (NaNO_3). Potassium nitrate (KNO_3).

Method. — See textbook, page 289.

Take special care to have test tubes and glass rods used in this work perfectly clean.

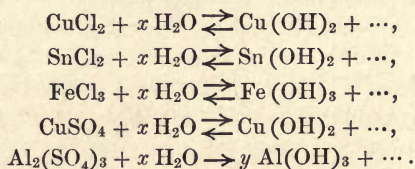
NOTE: — Before doing the work, read up the subject of hydrolysis in the textbook. In testing with litmus paper, put a small drop of the liquid on the paper with a glass rod. Use the same paper for many tests. In the interpretation of the results, remember that the reddening of blue litmus is positive proof of the presence of an acid

(that is, of hydrogen ions H^+), since nothing else can produce the change in color. The turning blue of red litmus is proof of the presence of a base (that is, of hydroxylions OH^-). Absence of effect on both kinds of litmus shows, therefore, that neither acids nor bases (H^+ and OH^- ions) are present in measurable quantity.

(a) Dissolve 0.2 c.c. of each of the substances mentioned in (a) in 10 c.c. distilled water in a small test tube. Use a separate tube for each substance. Heat until solution is complete. Then cool in a stream of water.

(1) Are the liquids completely clear? If the eye alone does not detect evidence of the formation of a precipitate, a very delicate test is to focus a beam of sunlight in the liquid with a lens. The smallest traces of solid matter make the path of the beam visible, on the same principle as the motes in a sunbeam. Try this with each solution. Try it also with the distilled water used, for comparison.

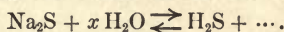
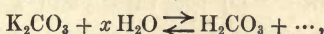
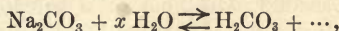
(2) Ascertain the action of each liquid upon red and blue litmus paper. What is, in each case, the only acid which could possibly be present? What, then, must be the other product? Assuming that you detected evidence of the formation of precipitates in (1), what is probably the nature of the precipitate in each case? Complete the following equations, supplying coefficients where they are denoted by letters:



The five substances in (a) are salts of *active* acids and *inactive* bases. Explain what this statement means. Do your results corroborate it?

(b) Treat the substances mentioned in (b) in the same way. Heating and the examination for suspended solid matter may be omitted, since the acids and bases formed by hydrolysis of

these three salts are all freely soluble. How do these substances differ from those in (a)? What is the only base which could possibly be present in each solution? What, then, must be the second product in each case? These three substances are salts of *inactive* acids with *active* bases. Explain the meaning of this statement and its bearing upon your results. Complete the equations: —



(c) Investigate the four substances in (c) in the same way. For the same reasons as in (b), the testing for separated solid matter can be neglected. How does this class of substances differ from the other two? Is there reason for assuming any interaction between the salts and water? These four substances are salts of *active* acids with *active* bases. What does this mean and what bearing has it upon your results? ¹

EXERCISE 62²

SEPARATION OF LEAD, SILVER, AND MERCURY

Apparatus.—Small test tubes. Beaker. Funnel. Glass rod. Wire gauze.

Materials.—Solutions of lead nitrate, silver nitrate, and mercurous nitrate. Potassium chromate solution (1:20). Filters. Sheet copper. Blue litmus paper.

Method.—See textbook, page 233 (table).

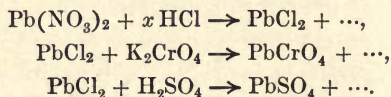
(a) (1) To 5 c.c. of lead nitrate solution in a small test tube, add dilute hydrochloric acid until the action is complete. Test this by shaking vigorously, letting the precipitate settle,

¹ If the time allotted to the subject permits, a classroom discussion of these phenomena from the standpoint of the ionization hypothesis would be valuable.

² With the usual laboratory period (ninety minutes) the teacher may find it best to take up (a), (b), and (c) one week and (d) and (e) the next.

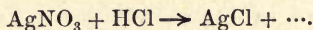
and adding a drop or two of hydrochloric acid to the liquid. Pour off the liquid as completely as you can without losing the precipitate, fill the tube half full of cold water, shake, and again pour off. What is removed by this treatment?

(2) Dissolve the precipitate in the smallest possible quantity of hot water. At once divide the solution into three equal portions. Stand the first aside and examine it when cold. Result? To the second, add 2 c.c. of a solution of potassium chromate. Result? To the third, add 1 c.c. of sulphuric acid. Result? Complete the equations:—



(b) (1) Treat 5 c.c. silver nitrate solution exactly as in (a) (1).

(2) Test the solubility of silver chloride in hot water. Pour off the water and divide the precipitate into two portions. Stand the first in the brightest light available for ten minutes. Meanwhile, test the action of ammonia on the second portion. Add *dilute* nitric acid to the ammonia solution until the odor of ammonia disappears. Result? Complete the equation:—



(c) Treat 5 c.c. of mercurous nitrate solution as in (a) (1). Divide the precipitate into two portions. Does hot water dissolve mercurous chloride? What effect has ammonia upon it?

(d) Mix 5 c.c. each of the solutions of lead, silver, and mercurous nitrates. Add dilute hydrochloric acid until it forms no further precipitate. Shake well and filter. Pour a little cold water over the precipitate. Reject the filtrate. What substances are contained in the precipitate?

(1) Wash the precipitate with 50 c.c. boiling water, being sure to reach every part of it. Preserve the liquid. Which of the three chlorides does it contain? Prove by using the test with potassium chromate and that with sulphuric acid as in (a) (2).

(2) What substances are still on the filter? Pour ammonia over it. Which dissolves? Receive the liquid in a clean test tube and add *dilute* nitric acid to it until the ammonia is neutralized (litmus paper).

(3) Which chloride has caused the darkening with ammonia? Make a little aqua regia by pouring 1 c.c. hydrochloric acid (concentrated) into 0.5 c.c. concentrated nitric acid. Warm it and pour it over the filter, receiving the filtrate in a clean test tube. Dilute with 10 c.c. water and place a 1×2 cm. (0.4×0.8 in.) piece of clean sheet copper in the liquid. Let stand. Wash, rub with the finger, and examine.

(e) Ask for an unknown solution and analyze it for silver, mercury, and lead, proceeding systematically as in (d). Note that the metals must be separated from each other before the tests can be applied. In writing your notes, remember that to prove the *absence* of a substance is frequently just as important (e.g. in cases of suspected poisoning) as to prove its presence.

EXERCISE 63

THE BEAD TESTS¹

Apparatus. — Platinum wire (mounted in piece of glass rod). Blow-pipe. Wing top.

Materials. — Borax powder. Microcosmic salt (sodium ammonium phosphate).

(a) Cobalt nitrate. Nickel nitrate. Manganese dioxide. Chrome alum. Copper sulphate. Ferric chloride. Sodium carbonate.

(b) Orthoclase (0.5 mm. fragment).

(c) *Optional.* — Siderite, rhodonite, chrysocolla.

Method. — See textbook, page 338.

Either the Bunsen burner or the blowpipe may serve as the source of heat. If the burner is used, reduce the access of air until a *small* yellow area appears in the upper portion of the flame. This luminous area contains glowing carbon. It is called the reducing flame. The

¹ Many teachers will prefer to inform the students beforehand what colors will be obtained in the beads by correct work.

strongest oxidizing flame is the extreme upper part (blue), but any part of the outer mantle can be used. Clamp the burner at an angle of 25° with the vertical to avoid dropping substances into the chimney.

If the blowpipe is employed, wash off the mouthpiece. Use the wing top. Re-read the directions in Exercise 6. The *reducing flame* is obtained by placing the tip of the blowpipe just inside of the base of the edge of the flame and bending down the flame with a gentle blast. It should be somewhat yellow and perfectly steady and noiseless. It should surround the sample completely. *In all blowpipe work the air-holes at the base of the burner should be closed.*

It is convenient to assign borax and microcosmic salt to different groups of students.

Bend the end of the platinum wire into a circle 2 mm. (0.08 in.) in diameter. Heat the loop red hot and dip it into powdered borax. Notice the swelling up due to loss of water of crystallization. Melt the anhydrous borax to a bead. Touch with the hot bead a *speck of cobalt nitrate not more than 0.5 mm. in diameter*. Return to the flame. When the bead appears clear, let cool and examine. If the bead is opaque, too much cobalt nitrate was used. Discard it and make another. Much more rarely it happens that the color is pale and indistinct. In that case, heat again and take up an additional speck of cobalt nitrate. Use the oxidizing flame in melting the bead. Then hold it persistently in the reducing flame and see whether the color changes.

Remove the bead by heating it red hot and dipping into water. The fragments can be detached from the wire without difficulty. Make a fresh bead and remove it, to cleanse the wire from cobalt, before proceeding to the next test. If the third bead has a color, discard it also. A clean, colorless bead should be used for each test.

In the same way, investigate the behavior of the substances mentioned in (a), each time studying the behavior in both flames. The result, with all except the last, is due to the metal of the salt. Any other compounds of the same metals would behave in the same way. In several cases, the bead

while hot (not red hot) has a different color from that which it has when quite cold. Does sodium carbonate give any color to the bead? Notice the escape of carbon dioxide in this case. This is the blowpipe test for a *carbonate*.

Record your results in a table thus:—

NAME OF COMPOUND	FORMULA	COLOR OF BEAD O. F. ¹		COLOR OF BEAD R. F. ¹	
		Hot	Cold	Hot	Cold

(b) If there is time for it, the substances in (a) may be tested in beads made with microcosmic salt, in exactly the same way.

In any case, make, with a clean bead from microcosmic salt and a 0.5 mm. bit of orthoclase, the *test for a silicate*. The insoluble residue which swims in the bead has the same composition as quartz (SiO_2). It is called the "silica-skeleton."

(c) is a suggested list of three minerals in which you are to identify the metal by the borax bead color in oxidizing and reducing flames. Also determine whether the substance is a silicate or carbonate.

¹ The abbreviations O.F. and R.F. are used for oxidizing flame and reducing flame.

EXERCISE 64¹

DYEING

Apparatus.—Seven agate pans. Liter flask for roughly measuring water. Glass rod. Trip scales.

Materials.—Cotton cloth (white). Flannel (white). Congo red. Aniline red. Malachite green. Distilled water. Sodium carbonate. Sodium sulphate. Tannic acid. Tartar emetic. Large gummed labels (Dennison's, No. 201). Paste.

Method.—See textbook, page 234.

A

Preparation of the cloth.—Cut into strips 4×2 cm. (1.6×0.8 in.). Each student will need 2 pieces of the flannel and 5 of the cotton. The sizing must be removed from the cotton by boiling it in an agate pan with a mixture of 40 c.c. concentrated hydrochloric acid and 1 liter tap water, rinsing under the hydrant and then dipping in a cold mixture of 10 c.c. ammonia and 1 liter tap water. This treatment is not necessary with the flannel.

B

Prepare the following baths:—

(1) 1 gram *Congo red* is dissolved by stirring in 1 liter of distilled water in an agate pan. Add 1 gram sodium carbonate and 2 grams sodium sulphate.

(2) 0.5 gram *aniline red* in 1 liter distilled water.

(3) 0.5 gram *malachite green* in 1 liter distilled water.

(4) 1 gram *tannic acid* in 1 liter distilled water.

(5) 1 gram *tartar emetic* in 1 liter distilled water.

(1), (2), (3), and (4) are heated to gentle boiling. (5) should be cold. Each pan should be plainly labeled with its number.

¹ In order to finish in a laboratory period, it will be necessary to distribute the preparation of the baths to different students.

C

(a) Wet a strip of cotton cloth, place it in (1) (gentle boiling), and move it about with a glass rod for one minute. Remove and wash well under the tap. Congo red belongs to the class of dyes which dye cotton *without a mordant*. Fasten the dyed cloth to your notebook page.

(b) Place a second strip of cotton cloth in (2) and a third in (3) for one minute. Remove and wash well. Result? Aniline red and malachite green are examples of a great class of dye-stuffs which do not give fast colors on cotton without the use of a mordant.

(c) Boil two strips of cotton cloth in (4) for five minutes. Wring out and keep in motion in (5) for five minutes. Now place one strip in (2) and move about constantly for one minute. Treat the other similarly in (3). Try to wash out the color. Result? What part is played by the mordant (4 and 5)? Mount the dyed cloth on your notebook page.

(d) Stir a strip of flannel in (2) and a second in (3) for one minute, without mordanting. Remove and try to wash out the color. What difference do you discover in the action of wool and of cotton toward dyes of the class to which aniline red and malachite green belong? Mount the dyed cloth on the notebook page.

Thoroughly wash the pans, using a little concentrated hydrochloric acid, if necessary, to remove the dyestuff.

EXERCISE 65

NITRIC ACID, AQUA REGIA

Apparatus. — Retort (150 c.c.) with glass stopper. Agate pan. Beaker. Small test tubes. Trip scales. Glass rod. Mortar and pestle. Manila paper.

Materials. — Sodium nitrate. Mossy zinc. Magnesium ribbon. Iron filings. Tin foil. Colored cloth of any kind. Gold leaf. Ferrous sulphate. Manila paper.

Method. — See textbook, page 297.

CAUTION: *The gases given off when nitric acid acts upon metals and the vapor of nitric acid itself are poisonous and must not be inhaled. Do not get nitric acid upon the skin or clothing.*

(a)¹ Fit up the apparatus shown in Fig. 21. Roughly weigh on the trip scales enough sodium nitrate to fill the retort about

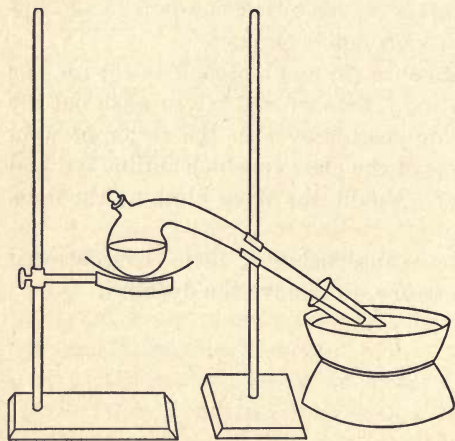


FIG. 21.

one third. Weigh off in a beaker an equal quantity of concentrated sulphuric acid. Introduce the sodium nitrate into the retort by means of a piece of paper. Add the sulphuric acid, insert the *glass stopper* (not a rubber or wooden cork, which would be rapidly destroyed by the nitric acid), and heat gently. Only vapor, not solid or liquid, must pass over.

Why? Empty the liquid which remains in the retort into the

¹ If the period is short, it may be necessary to omit or to postpone (b) and (c).

vessel provided for the purpose. (*Care!*) When the retort is cold, half fill it with water and boil gently to clean it.

Examine the nitric acid which collects and record its properties. Use the nitric acid in the following experiments. Cover a piece of zinc with water in a test tube and *slowly* add nitric acid. Do the same thing with a little iron filings and a piece of magnesium. In these reactions water is produced, together with the nitrate of the metal used. The gas produced may be nitrous oxide, nitric oxide, or nitrogen peroxide, according to the temperature and strength of the acid. Do not try to write the equations, which are quite difficult. Simply remember the general character of the action and the way in which it differs from the action of sulphuric or hydrochloric acid on the same metals (see textbook, page 298).

Drop a fragment of tin into concentrated nitric acid in a test tube. The metal is converted into tin oxide by the oxygen of the nitric acid. Let a drop of nitric acid fall upon a colored fabric of any kind.

(b) **Aqua regia.** — Pick up a small piece of gold leaf with the end of a wet glass rod, and rinse it into a test tube with water. In the same way place another piece of gold leaf in another test tube. Add to one tube nitric acid and to the other hydrochloric acid. Heat both tubes. Does the gold dissolve? Pour the contents of either tube into the other, and go on heating. (?) The product is gold chloride, and the gold dissolves because the oxygen of the nitric acid liberates chlorine from the hydrochloric acid and the chlorine attacks the gold. The mixture of nitric and hydrochloric acids will also dissolve platinum.

(c) **Test for nitric acid or a nitrate ($\overline{\text{NO}_3}$ ions).** — *Exercise unusual care in following directions.* — Select 2 c.c. of clean ferrous sulphate crystals, wash in running water, dry with paper (manila), and powder in a clean mortar. Shake the powder with 10 c.c. water (no heat) until you have a strong solution.

Place 3 c.c. of the clear liquid in a small test tube, incline

the tube at an angle of 45° , and allow 5 c.c. strong sulphuric acid to trickle slowly down the side. Avoid shaking. Being denser, the acid will form a layer below the solution.

Now, add in the same way, a few drops of nitric acid which has been diluted with twenty times its volume of water. Result?

Repeat, using a few drops of sodium nitrate solution about 1 : 20.

If time permits, repeat, using other nitrates (*e.g.* potassium nitrate, ammonium nitrate) to show that the test is for $\overline{\text{NO}}_3$ ions in general, not for any special nitrate.

EXERCISE 66

POTASSIUM NITRATE

Apparatus.—Evaporating dish. Glass plate. Funnel. Beaker. Lens. Wire gauze. Trip scales. Glass rod. Graduated cylinder.

Materials.—Sodium nitrate. Potassium chloride. Potassium nitrate crystals. Silver nitrate solution. Distilled water. Filters.

Method.—See textbook, page 301.

Place in a dish 25 c.c. water. Add 12 grams sodium nitrate and 11 grams potassium chloride. Heat on wire gauze, stirring with a glass rod until both are completely dissolved.

Stir gently and continue heating until half the water has evaporated (small flame). Pour off the clear liquid into a beaker and let it stand until cool. Meanwhile, scrape the crystals left in the dish into a filter supported in a funnel. Press carefully with the rod so as to squeeze out as much of the liquid as possible. Put a few crystals on a glass plate and examine with a lens or low-power microscope. What is the form? Only four substances might be obtained from your solution in this way. What are they? Which of them do you know to crystallize in the form you have observed (textbook, page 206). Taste the crystals (a trace only). Does the taste confirm your opinion?

Examine the crystals which have separated in the beaker and treat them in the same way. Compare them with the crystals of potassium nitrate in the stock bottle. Use the lens or, if necessary, the microscope.

Look up the solubility curves of sodium chloride and of potassium nitrate at different temperatures (textbook, page 207).

Why did the sodium chloride separate while the liquid was hot? Why did the potassium nitrate remain dissolved until the liquid cooled?

Does your potassium nitrate contain any sodium chloride as an impurity (look again at the solubility curve for sodium chloride)? Verify your prediction by dissolving 0.5 c.c. of your potassium nitrate in distilled water and testing the solution for chloride with one drop of silver nitrate solution. Reserve. Dissolve the rest of your potassium nitrate in 10 c.c. hot water and allow the solution to cool and crystallize. Test again with silver nitrate. Is the second crop of crystals purer than the first? Why?

EXERCISE 67

PREPARATION OF SODIUM NITRITE

Apparatus.—Iron dish. Beaker. Evaporating dish. Funnel. Glass rod. Two small test tubes. Trip scales.

Materials.—Sodium nitrate. Lead foil or test lead. Filters.

Method.—See textbook, page 305.

Melt 10 grams sodium nitrate in an iron dish and heat it strongly (take special care not to spill any of the melted substance upon the table, since it would burn deeply into the wood). Add gradually 20 grams of test lead (granulated) or the same weight of lead foil cut into small pieces. Stir with a glass rod and continue heating and stirring until the metal has almost completely disappeared. Let cool.

Meanwhile, boil 30 c.c. of water in a beaker and pour it

upon the cold substance in the iron dish. Boil for a short time and filter, receiving the filtrate in a porcelain dish. Evaporate carefully to small volume and let it crystallize. The product is sodium nitrite.

Compare it with sodium nitrate. Is there any difference in color? Dissolve 1 c.c. of each in 5 c.c. water and cautiously add 1 c.c. strong sulphuric acid. Results?

What is the purpose of adding the lead? What substance remained on the filter?

EXERCISE 68¹

NITRIC OXIDE

Apparatus. — Gas-generating bottle with thistle tube and delivery tube. Test tube. Agate pan. Iron spoon. Three bottles. Glass plates or filter paper for covering bottles. Watch glass. Beaker. Wire gauze.

Materials. — Copper clippings (No. 12 wire cut into 1 cm. pieces is suitable). Iron wire. Candles. Sulphur. Red phosphorus. Asbestos paper.

Method. — See textbook, pages 298, 299, 304.

CAUTION: Nitric oxide is poisonous. Keep the bottles of the gas covered during combustions and let the products escape out of the windows.

Nitric oxide is made in the same apparatus which is used for generating hydrogen. Fill the generator to the depth of about 1 cm. with copper clippings or cut pieces of sheet copper. In another bottle, dilute 50 c.c. nitric acid with twice its volume of water. Cool this liquid and pour it upon the copper until the generator is one third filled. Make sure that the thistle tube dips into the liquid. Collect the gas over water. It is well to stand the generator in cold water. If the evolution of gas becomes too energetic, pour a little water down the thistle tube. Collect three bottles and one test tube of the gas. Then stop, as even the first gas which comes off is not quite pure and the later portions contain large quantities

¹ Two students can work together to advantage.

of *nitrous* oxide and other impurities. Leave the bottles standing in the water.

Disconnect the apparatus (*do not inhale the gas*) and pour a little of the blue liquid into a watch glass. Use the watch glass as a cover for a beaker half filled with water kept gently boiling. Let the blue liquid evaporate *almost* to dryness. What is the product? Wash off the copper in the generator and return what is left of it to the stock bottle.

Use the test tube of the gas to investigate its solubility in water. Expose a bottle of nitric oxide to the air. Result?

Slip aside the cover of a bottle and plunge to the *bottom* of the bottle some sulphur burning vigorously in a spoon lined with asbestos paper. Remove the sulphur at once, keeping the bottle covered, and do the same thing with a lighted candle. Why should the candle be placed in the bottom of the bottle? Interpret the results.

Reline the spoon, fill it with red phosphorus, ignite, and plunge into a bottle of the gas. Result? Before putting away the spoon, heat it to redness to burn out any unused phosphorus.

EXERCISE 69¹

NITROUS OXIDE

Apparatus. — Large test tube with rubber stopper and delivery tube, connected as shown in cut. Four bottles. Two small test tubes. Agate pan. Iron spoon. Glass plates or filter paper for covering bottles.

Materials. — Ammonium nitrate. Christmas-tree candles. Iron wire. Sulphur. Red phosphorus. Wooden splints. Asbestos paper.

Method. — See textbook, page 303.

Fill a large test tube one third with ammonium nitrate. Clamp it at an angle of 45°, and insert a perforated stopper with a delivery tube. It is advisable, though not absolutely

¹ Two students can work together to advantage.

necessary, to pass the gas through an empty large test tube closed by a doubly perforated stopper before collecting it.

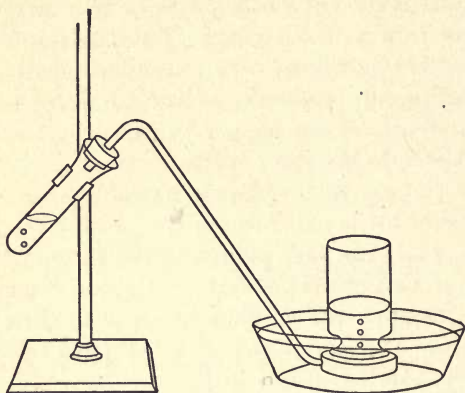


FIG. 22.

The apparatus is shown in Fig. 22. Apply a gentle heat and collect the gas over warm water. *The evolution of gas must be slow — a bubble or two a second. If it becomes too rapid, explosions result.* This can be easily controlled by lowering or removing the flame. Stop heating and remove

the stopper before the ammonium nitrate is exhausted. *Explosions sometimes occur when the quantity of substance becomes small.* If the ammonium nitrate shows signs of giving out before you have enough gas, disconnect, add more ammonium nitrate directly to the liquid in the test tube, and resume heating cautiously. Is there evidence of the formation of any product besides the gas? What?

Collect four bottles and two test tubes full of the gas. Cover the bottles with glass plates or wet filter paper, and stand them upright on the desk. Use one bottle to determine color, odor, and taste. Try the spark test in the test tube. Ascertain if the gas is soluble in water. Plunge a lighted candle into a bottle of it. Result? Set fire to some sulphur in a deflagration spoon lined with asbestos paper, and *at the instant the sulphur begins to burn* plunge it into the third bottle of nitrous oxide. It should be extinguished. Cover the bottle, and heat the sulphur until it burns vigorously. Plunge it again into the gas. Result? Is nitrous oxide easy or difficult to decompose into its elements? Which has probably

the higher temperature, the candle flame or the flame of burning sulphur? Remove the asbestos and reline the spoon. Fill it with red phosphorus, ignite, and plunge into a bottle of the gas. Result?

EXERCISE 70

PHOSPHATE FERTILIZERS

Apparatus.— Porcelain dish. Small test tubes. Glass rod. Funnel.

Materials.— Sodium hydrogen phosphate (Na_2HPO_4). Ammonium nitrate. Ammonium molybdate solution. Apatite or phosphate rock. Commercial superphosphate. Filters.

Method.— See textbook, page 331.

(a) Dissolve in a small test tube 0.5 c.c. sodium hydrogen phosphate and 1 c.c. ammonium nitrate in 5 c.c. water, and add five drops of nitric acid. Add 2 c.c. ammonium molybdate solution and warm gently, not to boiling. Result? The composition of the product depends upon the temperature, so that no formula can be assigned to it.

This is the *test* for phosphoric acid, or a soluble phosphate (that is, for $\overline{\text{PO}}_4$ ions).

(b) Place 10 grams powdered apatite or phosphate rock in a dish and add 4 c.c. water and 4 c.c. concentrated sulphuric acid. Warm for ten minutes, stirring with a glass rod. Add 10 c.c. water, warm, filter, and test the clear liquid as in (a), omitting the sodium hydrogen phosphate. Has the action of the sulphuric acid on the mineral produced a soluble phosphate? Explain (see textbook, p. 331).*

(c) Clean the dish, place 2 c.c. superphosphate in it, add 50 c.c. water, and boil. Filter and test as in (b). Result? Does the fertilizer contain a soluble phosphate? What is the object of the sulphuric acid in its manufacture?

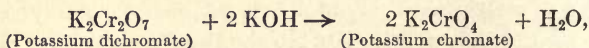
EXERCISE 71¹POTASSIUM CHROMATE AND POTASSIUM
DICHROMATE

Apparatus. — Graduated 100 c.c. cylinder. Trip scales. Beaker. Evaporating dish. Three small test tubes.

Materials. — Potassium hydroxide. Potassium dichromate. Potassium chromate. Dilute solutions of barium chloride, lead nitrate and silver nitrate. The potassium dichromate should be powdered to hasten solution.

Method. — See textbook, pages 342 and 343.

(a) Calculate the molecular weight of potassium dichromate. Using decigrams as your unit, weigh off one fourth of the molecular weight and dissolve it in 50 c.c. water in a dish, using gentle heat, if necessary. From the equation —



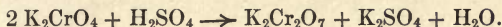
calculate the quantity of potassium hydroxide which would be required to interact with the potassium dichromate. Weigh off this quantity, dissolve it in 10 c.c. water, and add it to the solution of the dichromate. What does the color change indicate?

Estimate roughly how far the solution must be evaporated in order to deposit three fourths of its potassium chromate on cooling. Calculate the quantity of potassium chromate from the equation above. Assume that 1 c.c. water dissolves two thirds of a gram of potassium chromate at room temperature.

Evaporate the solution as far as you think it is necessary and let it cool. Meanwhile, go on with (c). If you do not obtain a good crop of crystals, evaporate further and find the error in your calculation. Examine the crystals and compare them with potassium dichromate.

¹ If the laboratory period is short, two students can work together to advantage.

(b)¹ Your dish must contain one half the molecular weight of potassium chromate, measured in decigrams. Why? Calculate the quantity of sulphuric acid required to interact with this according to the equation: —



Dissolve the potassium chromate in 50 c.c. water and slowly add the calculated quantity of sulphuric acid, stirring. What does the change in color indicate? If 8 parts water dissolve 1 part potassium dichromate at room temperature, how far must you evaporate in order to allow three fourths of the substance to crystallize on cooling? To answer this, you must know the amount of potassium dichromate in the liquid. Is it necessary to calculate this? Why? Evaporate to the volume estimated and let the solution crystallize.

(c) Dissolve 1 c.c. potassium dichromate in 20 c.c. water. In three separate test tubes (small), investigate the behavior of the solution with solutions of barium chloride, lead nitrate, and silver nitrate. Use not more than 5 c.c. of each solution.

The precipitates are the chromates of the corresponding metals. Record their properties and see whether they dissolve in nitric acid.

¹ If the laboratory period is short, (b) can be assigned to a second student and started simultaneously with (a). Use one half the molecular weight of potassium chromate, taking decigrams as the unit.

EXERCISE 72

AMMONIUM DICHROMATE

Apparatus.—Four small test tubes, one of which must be dry. Glass rod.

Materials.—Solid ammonium dichromate $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$. Solutions of barium chloride, lead nitrate and silver nitrate. Potassium hydroxide solution. Red litmus paper.

Method.—*The chemical behavior of a substance when ionized and when not ionized is very different.*

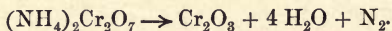
(a) Repeat (c) of the preceding exercise, using ammonium dichromate instead of the potassium salt.

To 5 c.c. ammonium dichromate solution add ammonium hydroxide until the color change is complete. Does it behave like potassium dichromate? What must have been formed? Equation?

(b) To 5 c.c. ammonium dichromate solution add potassium hydroxide solution until the color is pure yellow. Then add as much more potassium hydroxide and heat gently. Note odor. Test the gas with red litmus paper. With a rod wet with concentrated hydrochloric acid. What is it?

Ammonium dichromate in water solution is largely ionized to NH_4^+ , NH_4^+ , and $\text{Cr}_2\text{O}_7^{2-}$. Accordingly, like other salts it yields two classes of reactions, those of the ion NH_4^+ , of which (b) is an example; and those of the ion $\text{Cr}_2\text{O}_7^{2-}$, of which various examples are met in (a).

(c) Now investigate the conduct of the un-ionized substance. Place 1 c.c. of the crystals in a dry test tube and heat gently. Stop heating when the action begins, but resume heating, if necessary, to complete it. Hold the tube with a paper holder or a clamp. The chief products are nitrogen, water (steam), and chromic oxide (solid).



Note that this decomposition is totally different from the reactions of the same substance in water solution. In the

latter case it behaves as though the NH_4 and Cr_2O_7 had parted company and gives a different set of reactions for each, much as a solution containing both copper and silver ions will give the tests for both metals. In (c) there is no evidence of this separation. The formation of water indicates, for instance, that the hydrogen and the oxygen are in the same molecule.

Examine the chromic oxide carefully and test its solubility in hydrochloric acid.

EXERCISE 73

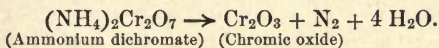
THE PERCENTAGE OF CHROMIUM IN AMMONIUM DICHROMATE

(Quantitative)

Apparatus.—Porcelain crucible and cover. Pipestem triangle. Balance.

Material.—Ammonium dichromate.

Method.—When ammonium dichromate is heated, the following change occurs:—



The percentage of chromium in the sample can be calculated from the weight of the chromic oxide left in the crucible.

Clean and weigh crucible and cover. Place cover on balance pan beside crucible and weigh in the crucible 0.5 gram ammonium dichromate. Cover crucible, place it on the triangle, and heat cautiously with a small flame kept in motion. Hold down the lid for the first few minutes by placing the tip of your pencil through the ring. If any noise is heard or any smoke appears, stop heating for a moment until quiet is restored. Finally remove pencil and apply the full heat of the burner for ten minutes; cool and weigh *without removing cover*.

Calculation.—The weight of the chromium in your sample is obtained by working out the following fraction:—

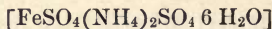
$$\frac{\text{weigh chromic oxide} \times \text{twice the atomic weight chromium}}{\text{molecular weight chromic oxide}}.$$

The percentage of chromium is:—

$$\frac{\text{weight chromium} \times 100}{0.5}.$$

EXERCISE 74

THE PERCENTAGE OF IRON IN FERROUS AMMONIUM SULPHATE

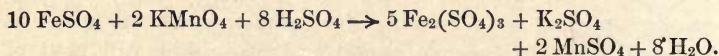


(Quantitative)

Apparatus.—Burette. Beaker. Stirring rod (thin, 3 mm. diameter. Thick rods cause breakage of beakers). Graduated cylinder. Balance.

Materials.—Ferrous ammonium sulphate. Distilled water. A solution of potassium permanganate, KMnO_4 , of such strength that 1 c.c. corresponds to 0.01 gram of iron. This solution is made by dissolving 5.643 grams of potassium permanganate in water and diluting the solution to 1 liter in a flask with a mark on the neck. The student can prepare the solution by dissolving 0.564 gram of potassium permanganate in 50 c.c. distilled water in a beaker. Make sure that the permanganate is *completely dissolved*. Pour into a graduated cylinder, and wash the beaker with 5 c.c. distilled water, which is poured into the cylinder. Repeat the washing three times. Dilute with distilled water to exactly 100 c.c. and *mix thoroughly*.

Method.—In presence of sulphuric acid, potassium permanganate converts ferrous sulphate, FeSO_4 , into ferric sulphate, $\text{Fe}_2(\text{SO}_4)_3$, thus:—



Weigh in a dry, clean beaker 2 grams of ferrous ammonium sulphate, half fill the beaker with distilled water, and stir until

the solid is *completely* dissolved. Do not heat. Add 3 c.c. concentrated sulphuric acid.

Fill the burette to zero with the permanganate and stand the beaker on a piece of white paper. Slowly run in the permanganate, stirring constantly. When the purple color seems to spread through the liquid in the beaker before disappearing, you are near the end, and the permanganate should be added one drop at a time; stirring after each. The *first perceptible* color which remains after stirring a few seconds indicates that the change is complete. If in doubt, record the reading and add another drop. If the color becomes more intense, the previous reading was the end.

Calculation.—The reading of the burette $\times 0.01$ = weight of iron in sample.

$$\frac{\text{weight of iron in sample} \times 100}{2} = \text{percentage of iron.}$$

EXERCISE 75

DETERMINATION OF THE PERCENTAGE OF CHLORINE IN SODIUM CHLORIDE

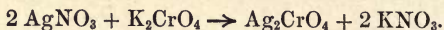
(*Quantitative*)

Apparatus.—Burette. Small test tubes. Beaker. Stirring rod (thin, 3 mm. diameter). Balance.

Materials.—Pure sodium chloride. Decinormal (one-tenth normal) silver nitrate solution (made by dissolving 17 grams silver nitrate in distilled water and diluting to 1 liter in a volumetric flask). The student can prepare the solution by dissolving exactly 1.7 gram of silver nitrate in distilled water in a graduated cylinder, diluting to 100 c.c. and mixing thoroughly. Solution of potassium chromate (20 grams) in distilled water (1 liter). Potassium chloride and potassium bromide are optional.

Method.—Dissolve a crystal of sodium chloride in 5 c.c. distilled water in a clean test tube and add a few drops of silver nitrate solution. In another tube, mix 1 c.c. of silver nitrate solution with 1 c.c.

of potassium chromate solution. The red precipitate is *silver chromate*:—



Now when silver nitrate solution is dropped into a liquid containing *both* sodium chloride and potassium chromate, *silver chloride only* is formed as long as any chlorine ions remain. When the chlorine ions are all converted into silver chloride, the first drop of silver nitrate in excess gives the red color of silver chromate.

Weigh 0.2 gram of sodium chloride in a clean, dry beaker, *dissolve* in 50 c.c. distilled water, and add 2 c.c. potassium chromate solution. Fill the burette to zero with the silver nitrate. Stand the beaker on white paper and slowly run in the silver nitrate solution, stirring constantly. The *first faint permanent change in color* is the end. When in doubt, record the reading and add another drop.

Calculation.—

1 c.c. decinormal silver nitrate = 0.00355 gram chlorine.
and, hence, burette reading \times 0.00355 = weight of chlorine in sample;

$$\frac{\text{burette reading} \times 0.00355 \times 100}{0.2} = \text{percentage of chlorine.}$$

In the same way, determine the chlorine in potassium chloride, taking 0.3 gram.

In the same way, determine the bromine in potassium bromide, taking 0.4 gram.

1 c.c. decinormal silver nitrate = 0.008 gram bromine.

EXERCISE 76

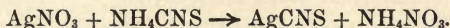
ANALYSIS OF A SILVER COIN

(Quantitative)

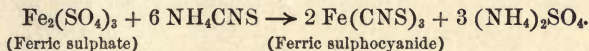
Apparatus.—Balance. Burette. Graduated cylinder. Beaker. Thin stirring rod. Watch glass. Test tubes. Tinner's shears.

Materials.—Silver nitrate. Silver nitrate solution. Ammonium sulphocyanide (NH_4CNS). Solution of ferric ammonium sulphate ($\text{Fe}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24 \text{H}_2\text{O}$), twenty grams to about 1 liter of distilled water. Distilled water.

Method.—Dissolve a crystal of ammonium sulphocyanide in 10 c.c. distilled water and divide the solution in two portions. To one portion add a few drops of silver nitrate. The white precipitate is silver sulphocyanide:—



To the other portion add a few drops of ferric ammonium sulphate. The red color is due to ferric sulphocyanide, formed from the ferric sulphate and the ammonium sulphocyanide:—



Now when ammonium sulphocyanide is added slowly to a liquid containing *both* silver nitrate and ferric sulphate, silver sulphocyanide *only* is formed until the silver is all precipitated and then the first drop of ammonium sulphocyanide in excess gives the liquid the red color of ferric sulphocyanide and indicates that the end has been reached.

In a graduated cylinder, dissolve 0.7 gram ammonium sulphocyanide in 100 c.c. distilled water and mix thoroughly. 0.315 gram of pure silver nitrate contains just 0.2 gram silver. Weigh off this quantity of silver nitrate in a clean, dry beaker, *dissolve* it in 50 c.c. distilled water, add 1 c.c. nitric acid and 1 c.c. ferric ammonium sulphate solution.¹ Fill the burette to zero with the sulphocyanide solution, stand the beaker on

¹ If the laboratory period is short, the instructor may prefer to prepare a large quantity of the sulphocyanide solution and standardize it beforehand. The experiment then requires only about 30 minutes.

white paper, and allow the sulphocyanide to run in until the color change occurs. Stir constantly and add the sulphocyanide in small quantities. Endeavor to hit the first perceptible change. When in doubt, take the reading and add another drop. Plainly:—

$\frac{0.2}{\text{burette reading}} = \text{weight of silver corresponding to 1 c.c. of your sulphocyanide.}$

Take about one tenth of a dime for the analysis. The coin can be easily cut with tinner's shears. Weigh the bit of coin accurately, place in a beaker, and add 2 c.c. distilled water and 2 c.c. nitric acid. Cover with a watch glass, place on wire gauze and put a *small* flame underneath.

When the coin is dissolved, add 50 c.c. distilled water and 1 c.c. ferric ammonium sulphate solution.

Fill the burette to zero with the sulphocyanide and proceed exactly as you did in standardizing your solution.

Calculation.—

$\frac{\text{burette reading} \times \text{silver value of solution} \times 100}{\text{weight of sample}} = \text{per cent of silver in coin.}$

EXERCISE 77

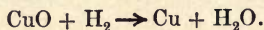
THE ATOMIC WEIGHT OF COPPER BY REDUCING CUPRIC OXIDE

(Quantitative)

Apparatus.—Hard glass test tube about 10 × 1.2 cm. (4 × 0.5 in.). Glass tube about 20 cm. (8 in.) long. Rubber tubing for connecting glass tube to gas supply. Balance.

Material.—Cupric oxide (this should have been heated to redness and placed in a stoppered bottle).

Method.—Cupric oxide when heated in hydrogen or illuminating gas is converted into copper:—



The atomic weight of copper is obtained from the loss in weight.

The hard glass tube must be clean and dry. Weigh it accurately. Cut a strip of paper 20×1 cm. (8×0.4 in.) and crease it in the middle to make a V-shaped trough. Use this to introduce the cupric oxide. Avoid getting the latter on the sides of the tube. Weigh in the tube about 1 gram of cupric oxide. It is not necessary to take exactly 1 gram, but whatever quantity you take must be accurately weighed.

Connect the glass tube by means of the rubber tube to the illuminating gas supply and, after lighting the gas, turn it down till you have a flame about 15 mm. (0.5 in.) high. Blow out the gas and set up the apparatus as shown in cut (Fig. 23).

Place the clamp 2 cm. (0.8 in.) from the mouth of the tube so that you may heat the cupric oxide without burning the clamp. Light the gas at the mouth of the test tube. Now heat the cupric oxide carefully and keep it at a dull red heat for five minutes. Let cool slightly and, by means of a towel, turn the tube until the cupric oxide is uppermost. Tap with a pencil until the cupric oxide falls. Heat again to redness for five minutes.

Remove the burner for five minutes; then extinguish the flame at the mouth of the tube; let cool and weigh. Place the copper in the receptacle provided.

Calculation. — The loss in weight is oxygen. The weight of the copper is obtained by subtracting the weight of the empty tube from the weight after heating.

weight oxygen : weight copper :: 16 : atomic weight copper.

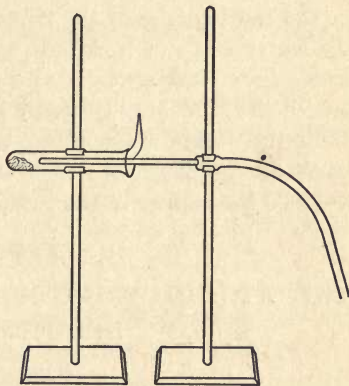


FIG. 23.

EXERCISE 78

PERCENTAGE OF COPPER IN COPPER SULPHATE
CRYSTALS*(Quantitative)*

Apparatus. — Same as in Exercise 77.

Material. — Copper sulphate, $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$.

Method. — Copper sulphate, heated in illuminating gas, leaves a residue of copper.

Use 1 gram of finely powdered copper sulphate and proceed exactly as in Exercise 77. Clamp the tube near the mouth and look out for the formation of water, which must be driven out by the cautious use of the flame. Have the tube horizontal, or the water will run back into the heated portion and produce breakage. Heat strongly at the last. The calculation may be left to the student. Compare your result with that calculated from the formula according to the method of the textbook, pages 89 and 385.

Place the copper in the receptacle provided.

EXERCISE 79

THE ATOMIC WEIGHT OF COPPER BY OXIDIZING
REDUCED COPPER*(Quantitative)*

Apparatus. — Porcelain crucible without cover. Triangle. Glass rod. Balance.

Materials. — Reduced copper from Exercises 77 and 78.

Method. — Although ordinary copper cannot be completely changed to oxide by heating, the reduced copper from Exercises 77 and 78 passes readily into cupric oxide at a red heat in the air.

Weigh the crucible and weigh in it from 0.5 gram to 1 gram of copper, which must previously be finely powdered in a mortar. Heat to redness for ten minutes. Let cool slightly and turn over with a glass rod. Heat again to redness for ten minutes; cool and weigh. The calculation is similar to that of the atomic weight of copper by reducing cupric oxide (Exercise 77).

EXERCISE 80

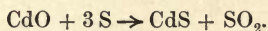
THE ATOMIC WEIGHT OF SULPHUR

(Quantitative)

Apparatus.—Porcelain crucible and cover. Triangle. Balance. Glass rod.

Materials.—Cadmium oxide. Flowers of sulphur.

Method.—Cadmium oxide when heated with sulphur passes into cadmium sulphide :—



From the increase in weight of the crucible, the atomic weight of sulphur can be calculated.

Weigh accurately a crucible without cover and weigh in it exactly 1 gram of cadmium oxide. Add a gram of sulphur roughly weighed. Since the excess of sulphur will be driven off by heat, the exact weighing of the sulphur would be useless. Mix well with a glass rod and dust off the rod into the crucible. Cover the crucible and place it on the triangle.

Turn the burner flame to full height and take the burner in the hand. Heat carefully, keeping the flame in motion and making sure that all parts of the crucible are heated, but not to redness. Especially avoid letting the bottom glow. See that the upper part, where the crucible and lid join, is thoroughly heated. Blue flames of burning sulphur appear here between lid and crucible. When they vanish, the heating is sufficient. *Do not heat too long.*

Calculation.—We start with the proportion :—

weight CdO : weight CdS :: mol. weight CdO : mol. weight CdS,

$$\text{mol. weight CdS} = \frac{\text{weight CdS} \times \text{mol. weight CdO}}{\text{weight CdO}}.$$

Since the atomic weight of cadmium is 112.5 and that of oxygen 16 we have :—

$$\text{atomic weight S} + 112.5 = \frac{\text{weight CdS} \times 128.5}{\text{weight CdO}},$$

$$\text{atomic weight S} = \frac{\text{weight CdS} \times 128.5}{\text{weight CdO}} - 112.5.$$

EXERCISE 81

THE FORMULA OF ZINC CHLORIDE

(*Quantitative*)

Apparatus. — Conical flask (capacity 80 c.c.). Asbestos plate (wire gauze can be used with care). Small watch glass. Balance.

Material. — Sheet zinc.

Method. — Zinc dissolves in hydrochloric acid to form zinc chloride, which can be obtained in anhydrous condition by evaporation.

Results are good only if the directions are exactly followed.

Weigh the flask, which must be dry, and place in it about 2 grams of zinc, accurately weighed. Add 10 c.c. concentrated hydrochloric acid, cover with a watch glass and heat gently with a small flame (which does not touch the asbestos plate) until the zinc is dissolved. Then increase the flame and boil the liquid until the water is evaporated. This is indicated by the fact that the formation of bubbles practically stops.

Now *reduce the flame until it does not quite touch the asbestos plate* and remove the watch glass. The zinc chloride will solidify and then melt. *Just as soon as it melts, stop heating and weigh when cold enough to handle.* Zinc chloride absorbs water from the air, and if the flask is allowed to stand long before weighing, it should be corked.

Calculation. —

You have the weight of the zinc: the weight of the zinc chloride is obtained by subtracting the weight of the flask from the final weight. The chlorine is, of course, the difference between the zinc and the zinc chloride.

Divide the weight of the zinc by the atomic weight of zinc, and that of the chlorine by the atomic weight of chlorine. You will obtain decimal fractions which will, however, be seen to bear to each other approximately the relation of small whole numbers. These are the numbers which must be added to the symbols to give the formula. An example of the calculation is given in the textbook, page 385.

EXERCISE 82

PERCENTAGE OF OXYGEN IN POTASSIUM CHLORATE

(*Quantitative*)

Apparatus.—Pipestem triangle. Porcelain crucible with cover. Balance. Small test tubes.

Materials.—Potassium chlorate. Potassium chloride. Silver nitrate solution. Distilled water.

Method.—The oxygen is driven off by heat and the loss in weight obtained.



Dissolve 0.2 c.c. potassium chlorate in 5 c.c. distilled water. In another tube, dissolve 0.2 c.c. potassium chloride in 5 c.c. distilled water. Add ten drops silver nitrate solution to each tube. Results? Potassium chlorate is apt to contain a trace of potassium chloride, so that a *slight* precipitate may be obtained in the first tube also. Weigh crucible, with cover, place the cover on the balance pan beside the crucible, and weigh in the crucible 1 gram of potassium chlorate. Heat carefully for ten minutes, keeping flame in motion; apply the full heat of the burner for five minutes more, cool and weigh without removing cover. Calculate the percentage of oxygen, and compare it with that calculated from the formula.

Examine the residue in the crucible. What is it? Dissolve it in water and add silver nitrate solution. Result?

EXERCISE 83

THE EFFECT OF HEAT AND OF ACIDS ON
CARBONATES

Apparatus.—Wire gauze. Hard glass test tube with one-hole rubber stopper and delivery tube bent at right angle. Evaporating dish. Glass rod. Small test tubes.

Materials.—Calcite (crystals or cleavage pieces). Magnesite. Malachite (powder). Commercial copper carbonate can be used instead of malachite. White lead. Lime. Red litmus paper. Lime-water.

Method.—Most carbonates are decomposed by heat, giving off carbon dioxide and leaving a residue of the oxide of the metal. Carbon dioxide escapes when carbonates are dissolved in acids.

(a) Select two small pieces of calcite, not more than 1 cm. (0.4 in.) long. Keep one for comparison. Support the other on wire gauze and heat it with the full power of the burner for ten minutes or more. Compare with the unheated piece. Look especially at the edges for a change. If no distinct alteration has occurred, continue heating until there is a visible result. Write the equation for the chemical change which has taken place.

Your equation, if correct, will call for the production of two substances, one of which is lime. Verify this as follows: Place a piece of red litmus paper in a clean dish and upon it a fragment of good lime 2 mm. (0.08 in.) in diameter. Moisten the lime with two drops of water, using a glass rod. Is there any evidence of chemical change? Write the equation. Now add enough water to moisten both lime and paper and wait several minutes. What does the change in the paper indicate?

Clean the dish, place your unheated crystal on a fresh piece of red litmus paper, and treat it in the same way. Is there any change?

Now treat the crystal which has been heated in exactly the same manner. Is your suspicion that lime is produced verified?

The second substance called for by your equation for the

heating of calcite is a gas. The difficulty about verifying its formation is that the temperature at which calcite decomposes is so high that a glass tube would be melted.

(b) *Magnesite* differs from calcite in containing magnesium instead of calcium. It behaves in the same way when heated except that the temperature required is not nearly so high. Select two pieces of it about 1 cm. in diameter. Reserve one for comparison. Heat the other in the hard glass test tube. The delivery tube dips into some clear limewater in a test tube. The heat should be gentle at first and the final temperature need not be high enough to soften the glass. Watch the limewater intently during the whole progress of the experiment. Take out the stopper before letting the hard glass tube cool.

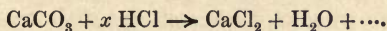
What gas escaped? Compare the magnesite which has been heated with the unheated mineral as regards appearance and cohesion. Compare the two in their behavior when placed on red litmus paper and moistened. Wait patiently for a result, as the action may be slow.

(c) *Malachite* is more complex because it is a *basic* carbonate. This means that it contains copper hydroxide as well as copper carbonate. Heat 1 c.c. of the powder in the same tube used for the magnesite, first wiping the tube out thoroughly with paper. The tube must be horizontal and the heat very gentle. The delivery tube dips into a fresh portion of limewater. There are three products. What are they and what is the evidence for their formation?

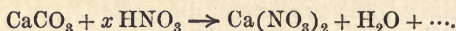
The red litmus test would be useless in this case.

(d) Heat 1 c.c. of *white lead* in the same way as malachite. Like malachite, white lead is a basic carbonate. Its formula is $\text{Pb(OH)}_2\text{PbCO}_3$. What three products are formed?

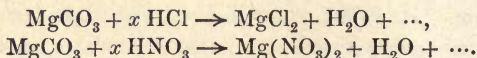
(e) Place a 2 mm. (0.08 in.) bit of calcite in a test tube, add 2 c.c. of water and 2 c.c. of concentrated hydrochloric acid. Result? What gas escapes? Complete the equation:—



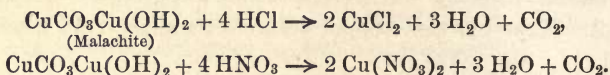
Repeat, using nitric acid instead of hydrochloric. Complete the equation:—



In the same way, investigate the interaction of magnesite and of malachite with hydrochloric and also with nitric acid. Use a gentle heat, if necessary. Complete the equations:—



In the case of malachite, the equations are more difficult and are, therefore, given in full:—



Make a general statement about the action of acids on carbonates. How could you ascertain whether a substance submitted to you was a carbonate or not?

EXERCISE 84

THE EFFECT OF HEAT ON A CARBONATE

(Quantitative)

Apparatus.—Porcelain crucible. Pipestem triangle. Small test tubes. Balance.

Materials.—Magnesium carbonate (powdered magnesite).

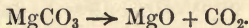
Method.—See Exercise 83.

(a) Heat, in a test tube, a mixture of 2 c.c. hydrochloric acid and 5 c.c. water nearly to boiling. Remove from the flame and add 1 c.c. powdered magnesite. Result? What gas escapes?

(b) Weigh a crucible without the cover and weigh in it a gram of fine magnesite powder. Heat gently at first, finally apply the full heat of the flame for fifteen minutes, cool and weigh. If time permits, the exactness of the result is increased

by reheating sharply for five minutes and weighing again to see if the decomposition is complete. Use the *lowest* weight obtained in your calculation.

The loss in weight is *carbon dioxide* : —



What is the *percentage* of carbon dioxide yielded by magnesite according to your results? How does your result agree with that calculated from the equation?

(c) Test the substance in the crucible by throwing it into a warm mixture of hydrochloric acid and water as in (a). Result?

EXERCISE 85¹

BORIC ACID AND BORAX

Apparatus.— Trip scales. Graduated cylinder. Beaker. Dish. Small dry test tube. Glass rod.

Materials.— Boracite. Boric acid. Borax. Sodium carbonate. Alcohol.

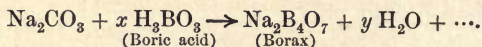
Method.— *Boric acid* is produced when borates are treated with active acids. *Borax* is obtained when boric acid or a borate is treated with a solution of sodium carbonate.

(a) Mix 10 grams powdered boracite with 10 c.c. concentrated hydrochloric acid and 10 c.c. water in a dish. Heat *gently* for ten minutes. Filter hot and let cool. The crystals are boric acid, H_3BO_3 . Boracite is chiefly magnesium borate. What, then, is the action of hydrochloric acid upon it? What magnesium compound must be contained in the liquid which covers the crystals?

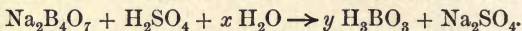
(b) Dissolve 12 grams boric acid in 60 c.c. of boiling water in a dish. Slowly add 10 grams anhydrous sodium carbonate. Recalling the behavior of carbonates with acids, what gas escapes? What must exist in the liquid? Evaporate until

¹ If the period is short, it may be convenient to distribute (a), (b) and (c) to different students.

the liquid is half gone and let cool. *Borax* separates. Compare the crystals with those of boric acid. Complete the equation:—



(c) Slowly add 10 c.c. concentrated sulphuric acid to 15 c.c. water. Pour this liquid into a hot solution of 10 grams borax in 50 c.c. water in a beaker. Let cool. Result? Complete the equation:—



(d) If time permits, test borax for water of crystallization by *gently* heating 2 c.c. from the stock bottle in a dry, clean test tube clamped in a horizontal position.

(e) In a clean dish mix 1 c.c. of borax with 0.5 c.c. of concentrated sulphuric acid. Add 2 c.c. alcohol and set fire to the liquid. Result? This is a *test* for boric acid or a borate.

EXERCISE 86

THE ATOMIC WEIGHT OF MAGNESIUM

Apparatus.—Wide-mouthed bottle holding about 400 c.c. Graduated 100 c.c. cylinder. Glass plate. Balance. Agate pan.

Material.—Magnesium ribbon.

Method.—Magnesium dissolves in hydrochloric acid:—



The atomic weight of magnesium can be calculated from the volume of the hydrogen liberated.

Weigh accurately not more than 0.4 gram of magnesium ribbon. The weight should be accurate to a milligram. If your balance is not exact enough for this, weigh 2 m. of the ribbon and calculate by proportion the length you must cut off in order to get the weight you desire to take. If your bottle holds less than 400 c.c., reduce the weight of magnesium proportionally, taking not more than 0.1 gram for each 100 c.c.

capacity. Coil the ribbon into a spiral, smaller than the mouth of the bottle.

Place 40 c.c. of concentrated hydrochloric acid in the bottle. Fill the bottle with water, adding the water carefully so as to allow the acid to remain at the bottom. With the aid of a glass plate invert the bottle in an agate pan of water. The water should be about 5 cm. deep in the pan. The bottle must not contain air bubbles. If a little air enters, withdraw the bottle carefully, using the glass plate, set it upright upon the desk, add water until it is completely filled, and try again.

Put the coil of magnesium ribbon in the pan, slide the mouth of the bottle (under water) over it and set it down.

What gas collects? What other substance must be formed and dissolve in the water?

When the action is over, add water to the pan or remove water, as may be necessary to make the level of the water inside and outside the same. Why? Tightly cover the bottle with the glass plate and remove it from the water, without allowing any water to escape from the bottle.

Set the bottle upright on the desk, strike a match, take off the glass plate, and instantly hold the burning match in the mouth of the bottle. Result?

With the graduated cylinder, measure (1) the water left in the bottle, (2) the total capacity of the bottle. Subtract (1) from (2) to obtain the volume of the hydrogen.

Calculation. — Subtract one fourteenth from this volume for *expansion*, due to the fact that the hydrogen is not measured at standard temperature. Multiply the remainder by one fiftieth and subtract to correct for the *water vapor*, which is measured with the hydrogen, since it is collected over water. The remainder will be approximately the volume which your hydrogen would occupy at STP.¹ Since the weight of 1 c.c. of hydrogen at STP is 0.00009 gram, the volume of your hydrogen in cubic centimeters multiplied by 0.00009 gives its weight in grams.

¹ STP means standard temperature and pressure, 0° and 760 mm.

The equation : —



shows that the atomic weight of magnesium in grams would set free two atomic weights or 2.02 grams of hydrogen. Hence, use the proportion : —

Weight in grams of your hydrogen : 2.02 :: weight in grams of magnesium taken : atomic weight of magnesium.

*Sample Calculation.*¹—A student found that 0.4 gram of magnesium produced 400 c.c. of hydrogen, measured under the conditions of the experiment. Since our measurements are made with a cylinder which does not read to fractions of a cubic centimeter, it is a waste of time to retain fractions of a cubic centimeter in the calculation.

$$400 \text{ c.c.} \times \frac{1}{1.4} = 29 \text{ c.c. (correction for expansion).}$$

$$400 \text{ c.c.} - 29 = 371 \text{ c.c.}$$

$$371 \text{ c.c.} \times \frac{1}{50} = 7 \text{ c.c. (correction for water vapor).}$$

$$371 \text{ c.c.} - 7 \text{ c.c.} = 364 \text{ c.c. (volume at STP).}$$

$$364 \text{ c.c.} \times 0.00009 = 0.03276 \text{ gram (weight of hydrogen).}$$

$$0.03276 : 2.02 :: 0.4 : x.$$

$$x = 24.66.$$

¹ This calculation is approximate, but the error does not exceed the probable error of the experimental measurement. The teacher may, however, prefer to calculate the result by the more exact methods explained in Chapter 30 of the textbook. A third method is for the teacher to calculate the weight of 1 c.c. of moist hydrogen, under the experimental conditions. This does not vary widely from 0.000082 gram. The student then calculates the weight of the hydrogen by multiplication and applies the proportion given above.

EXERCISE 87

THE PRODUCTION OF A DOUBLE SALT

Apparatus. — Two large test tubes. Three small test tubes. Beaker. Glass rod. Trip scales. Graduated cylinder. Lens. Funnel.

Materials. — Copper sulphate (commercial). Ammonium sulphate (commercial). Barium chloride solution. Lime. Filters. Iron nails.

Method. — When concentrated hot solutions of copper sulphate and of ammonium sulphate are mixed and allowed to cool, *copper ammonium sulphate* ($\text{CuSO}_4, (\text{NH}_4)_2\text{SO}_4, 6 \text{H}_2\text{O}$) is deposited in light blue crystals.

Using decigrams¹ as your unit, weigh off the molecular weight of copper sulphate ($\text{CuSO}_4, 5 \text{H}_2\text{O}$). Hold a large test tube nearly horizontal and place the copper sulphate in it. Add 30 c.c. water, clamp the tube at an angle of 45° , and place under it a *small* flame, just sufficient to heat the liquid to boiling.

Weigh off the molecular weight of ammonium sulphate $(\text{NH}_4)_2\text{SO}_4$ in decigrams, and treat it in the same way, using only 20 c.c. of water.

When both salts are *completely* dissolved, mix the solutions in a clean beaker and let cool. The double salt deposits. If no deposit forms, the solution is *supersaturated*, and it may be left as an exercise for the student to devise a method of causing crystallization to occur. Scrape the crystals on a filter, using a glass rod. Examine the double salt with a lens and compare it with copper sulphate and with ammonium sulphate. Dissolve 2 c.c. of the double salt in 15 c.c. hot water and divide the liquid into three portions in small test tubes. To one portion add a few drops of barium chloride solution. Result? Is the double salt a *sulphate*? To the second portion add an

¹ Although both sulphates are cheap, the teacher may prefer to use centigrams as a unit and one tenth the volume of water. Small test tubes should then be used.

iron nail and let stand. Result? To the third portion add 2 c.c. of lime and heat gently. Note odor of the gas which escapes. Test it with red litmus paper. Then hold in it a rod wet with hydrochloric acid. Result? What is proved by these three tests?

APPARATUS AND SPECIAL MATERIALS

The following list includes the apparatus and chemicals needed for the experiments in the Laboratory Studies.

Agate pan, round, 30×7.5 cm. (12×3 in.).

Asbestos boards, 10 cm. square.

Asbestos paper.

Balance — beam balance sensitive to a milligram, if possible, with weights from 50 g. to 1 mg. Such an instrument will cost about ten dollars, slightly more if purchased with case provided with sliding door and glass sides and back. Duty-free importation is assumed.

Balance — trip scales sensitive to a decigram with weights from 100 g. to 10 g.

Barometer.

Beakers, about 100 c.c. capacity.

Blowpipe, simplest form.

Bottles, common salt-mouth, about 400 c.c. capacity, no stopper.

Bottles, gas generating, 300 c.c.

Bunsen burner with wing-top.

Burette, Mohr form with rubber tube and pinchcock, 50 c.c., graduated to $\frac{1}{10}$ c.c. The Geissler burette with glass stopcock does not work well in the hands of a beginner.

Cigar lighters (tobacconists' splints) for spark test.

Clamps (burette), one for each stand.

Cobalt glass, 10 cm. square.

Corks to fit large and small test tubes.

Crucibles, porcelain, with cover, capacity 25 c.c.

Deflagrating spoon, iron, small.

Dennison labels, Nos. 201, 213, 223.

Dishes, iron, capacity 70 to 100 c.c.

Dishes, lead, capacity 50 to 100 c.c.

Dishes, porcelain evaporating, capacity 125 c.c.

File, small triangular.

Filter paper.

Filters, cut ordinary, 10 cm.

Flask, volumetric, 1 liter.

Flask, volumetric, 6 liters (optional, but very convenient).

Flasks, Erlenmeyer, 80 c.c.

Flasks, ordinary, 300 c.c.

Forceps, small steel, cheapest form.

Funnels, 75 mm. diameter.

Funnels, dropping, 80 c.c. capacity (optional, but convenient). A fair substitute can be made by cutting a thistle tube and uniting by a rubber tube bearing a clamp.

Glass plates 8 cm. square for covering bottles.

Glass rod, 3 mm. diameter.

Glass tubing, 7 mm. outside diameter.

Glass tubing, 14 mm. outside diameter, hard glass.

Graduated cylinder, 100 c.c. graduated to 1 c.c.

Hammer with heavy iron plate for crushing minerals.

Kipp generator, simplest form (optional).

Lens, Coddington, 3 cm. focus.

Magnet, small horseshoe.

Medicine dropper.

Meter stick, centimeters and millimeters on one side, inches and fractions on other.

Miner's safety lamp.

Mohr pinchcocks.

Mortar, 6 cm. porcelain, with pestle.

Platinum foil, 25 mm. square.

Platinum wire, B. & S. gauge, No. 25.

Pliers, gas, small.

Retort, 150 c.c. capacity, glass stopper.

Rubber bands (small).

Rubber stoppers, No. 0 (1-hole); No. 2 (1-hole); No. 3 (solid, 1-hole, 2-hole); No. 5 (1-hole, 2-hole).

Rubber tubing for gas connections, cloth impression, 8 mm. bore.

Rubber tubing for apparatus connections, heavy wall, 4 mm. bore. (The best grade of rubber stoppers and tubing should be purchased.)

Rubber tubing to renew clamps, cloth impression, 15 mm. bore.

Scissors, ordinary, for cutting paper, rubber tubing, etc.

Shears, tinner's, for cutting sheet metals.

Stand, about 150 cm. high, with one ring 7 cm. or 8 cm. in diameter.

Test tubes, 200 × 25 mm.

Test tubes, 150 × 18 mm.
Test tubes, 100 × 12 mm. (hard glass).
Thermometer, reading from -10° C. to 150° C.
Thistle tubes (small).
Waste jar, 4 gallon, earthenware glazed.
Watch glasses, 5 cm. and 8.5 cm.
Wire cutter (electrician's, small).
Wire gauze, iron, 10 cm. square.

CHEMICALS, ETC.

Acid, Acetic.	Cadmium Oxide.
Acid, Boric.	Calcium (metallic).
Acid, Formic.	Calcium Carbide.
Acid, Hydrochloric.	Calcium Carbonate (marble cracked in 1 cm. fragments).
Acid, Nitric.	Calcium Carbonate (precipitated chalk).
Acid, Oxalic.	Calcium Chloride (commercial dry).
Acid, Sulphuric.	Calcium Fluoride (powder).
Alcohol, 95 per cent grain.	Calcium Nitrate.
Alcohol, Wood Spirit.	Calcium Oxide (lime).
Aluminium (sheet or turnings).	Calcium Sulphate (powder).
Aluminium Sulphate.	Candles (Christmas-tree candles).
Ammonia Water.	Carbon (charcoal ordinary).
Ammonium Carbonate.	Carbon (charcoal, prismatic for blowpipe use).
Ammonium Chloride (commercial).	Carbon (charcoal powder).
Ammonium Dichromate.	Carbon Disulphide.
Ammonium Molybdate (solution).	Cardboard.
Ammonium Nitrate.	Chloroform.
Ammonium Sulphate.	Chrome Alum.
Ammonium Sulphocyanide.	Coal (bituminous).
Aniline Red.	Cobalt Chloride.
Arsenic, Metallic.	Cobalt Nitrate.
Arsenious Oxide.	Cobalt Sulphate.
Asbestos Fiber.	Cochineal.
Barium Chloride.	Congo Red.
Barium Nitrate.	Copper Sheet (ordinary).
Boneblack.	
Borax.	
Bromine.	

Copper Sheet, 0.05 mm. thick.	Manganese Dioxide (powder).
Cotton Cloth (colored).	Mercuric Chloride.
Cotton Cloth (muslin, white).	Mercuric Oxide.
Cupric Carbonate (basic).	Mercurous Nitrate.
Cupric Chloride.	Mercury.
Cupric Oxide.	Molasses.
Cupric Sulphate (commercial).	Nickel Nitrate.
Cupric Sulphate (C. P.)	Paraffin.
Cuprous Oxide.	Paste (photographic, in tube).
Emery paper (fine).	Phenol-phthalein.
Ether.	Phosphorus (red).
Ferrous Ammonium Sulphate.	Picture Cord.
Ferrous Sulphate.	Pins.
Flannel (white).	Potassium Alum.
Glucose.	Potassium Bromide.
Glue (solid).	Potassium Carbonate (commercial).
Gold Leaf.	Potassium Chlorate.
Hydrogen Peroxide.	Potassium Chloride.
Iodine.	Potassium Chromate.
Iron (powder by alcohol).	Potassium Cyanide.
Iron Alum (ferric ammonium sulphate).	Potassium Dichromate.
Iron Chloride (ferric).	Potassium Hydroxide.
Iron Filings.	Potassium Iodide.
Iron Nails.	Potassium Nitrate.
Iron Sulphide (ferrous).	Potassium Permanganate.
Kerosene.	Potassium Sulphate.
Lead (granulated, test lead).	Pyrogallol.
Lead Foil (test lead).	Rosin.
Lead Nitrate.	Sand.
Lead Oxide (litharge).	Sandpaper.
Lead, Red.	Silver Nitrate.
Lead, White.	Soda Lime.
Lithium Chloride.	Sodium.
Litmus Cubes.	Sodium Acetate (fused).
Litmus Paper (red and blue).	Sodium Ammonium Phosphate.
Magnesium Oxide.	Sodium Carbonate (dry).
Magnesium Ribbon.	Sodium Carbonate Crystals (commercial).
Magnesium Sulphate.	Sodium Chloride (C. P.).
Malachite Green.	

Sodium Chloride (common salt).	Tartar Emetic.
Sodium Hydrogen Carbonate.	Tin Chloride.
Sodium Hydrogen Phosphate.	Tin Foil (pure, free from lead).
Sodium Hydrogen Sulphate.	Thread.
Sodium Hydroxide.	Toluol.
Sodium Nitrate.	Vaseline.
Sodium Nitrite.	Water, Distilled.
Sodium Potassium Tartrate (Rochelle salt).	Wire, Copper (No. 12 B. & S. gauge).
Sodium Sulphate.	Wire, Copper (No. 20 B. & S. gauge).
Sodium Thiosulphate (commercial).	Wire, Copper (No. 30 B. & S. gauge).
Stannic Oxide.	Wire, German Silver (No. 36, B. & S. gauge).
Stannous Chloride.	Wire, Iron, No. 20.
Starch.	Zinc Dust.
Strontium Chloride (optional).	Zinc, Mossy.
Strontium Nitrate.	Zinc Oxide.
Sugar (ordinary).	Zinc (sheet).
Sulphur, Flowers.	Zinc Sulphate.
Sulphur, Roll.	
Superphosphate (fertilizer).	
Tannic Acid.	

MINERALS

Boracite.	Magnesite.
Calcite (cleavage pieces).	Orthoclase.
Chalcopyrite.	Phosphate Rock.
Chrysocolla.	Pyrite.
Cinnabar (10 per cent).	Rhodonite.
Galenite.	Siderite.
Gypsum.	Sphalerite.

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